chemicals to be locked down at any facility, under surveillance, and weighed regularly. They also expect anyone with access to List I chemicals to undergo a background check. The best advice I can give you is to forget about them, they're not worth whatever convenience in synthesis they might provide.

Finally, the last option for obtaining chemicals is the old fashioned, tried and true, five finger discount method. This differs from "the inside job" in that burglary, rather than shoplifting, is involved. Correspondingly, this is a much higher risk proposition. It is only advisable if one has inside information on the target regarding possible security on the site, and placement of the desired items. Information on the latter is openly available to all members of the community via the publicly filed "Emergency Planning and Community Right To Know" reports filed by businesses each year. A floor plan and location of chemicals is listed in the report. Call your county government for where the reports are kept. Say you're some environmentalist type or something similar. That will get you the floor plan and locations. Security measures aren't discussed in these reports, so be warned. Also be warned that the exact location of chemicals is only given for those contained within the plant above the reporting threshold. This is generally over a thousand pounds, but less for Extremely Hazardous chemicals. In any case, the floor plan is informative.

Chapter Two

The Fester Formula

The pressing need for someone to find and make public a simple, convenient, effective and clandestinely low profile method for producing stash amounts of meth has long been a matter of concern for me. A suitable method would have to pass several hurdles. To be suitable for the casual consumer-producer, the method would have to be operable without access to chemical glassware. This hurdle is higher than it would seem at first glance because it requires that the product produced from the process must be pure enough to use as is without distilling or other complicated manipulations. The method would have to be free of odiferous emanations which would alert meddlesome neighbors to unusual goings on. It's even better if the process is sufficiently odor free that it can be done in some room without disapproving wives or yakking children noticing. Yes, it would be really great if one could run a batch in a dorm room without anyone noticing. Finally, the chemicals used would have to be so common that anyone would have easy access to them, either at work or school, or even so common and possessing so many mundane uses that they can be safely ordered from any of the various mail-order "hobbyist" chemical suppliers without bringing any attention to the or-

derer. These hurdles have all been cleared in what is probably the greatest achievement of my career as a chemist — The Fester Formula.

Some background information will be of benefit to those who would wish to understand where the inspiration for this procedure came from. For some time now, it has been evident that the field of electro-organic synthesis is where clandestine chemistry should be moving. When doing an electric synthesis, a piece of metal and DC current are the reagents. The logistics of supplying oneself to do an electric synthesis are consequently quite simple. The only reason why this fertile field has not long ago been harvested by clandestine cookers is lack of education. One can easily go through four years of college chemistry, and even grad school, without ever being required to do an electric synthesis. Its techniques are, for some reason, not taught unless one chooses to go into the specialized field of electro-organic chemistry. The discoveries made by those working in this field are generally ignored by chemists in other fields, as they are quite comfortable using conventional chemical reagents to do their molecular manipulations. Even so, the electro-organic people are generally quite satisfied with the results they get, and their marginalization miffs them, to say the least.

It's my intent to plant a crop on this fertile ground, that you may reap the harvest. The strategy which has produced the most advances in clandestine chemistry is to take a general synthetic method, and apply it in a previously unknown manner. That's where the lithium metal in ammonia reduction of ephedrine or pseudoephedrine to meth or phenyl-propanolamine to Dexedrine recipe came from. Ditto for hydroiodic acid and red phosphorus put to the same purpose. Also, the Method X LSD synthesis using propionic anhydride is an extension of a previously known procedure.

A search of the chemical literature for electric methods of reducing benzyl alcohols, the class of chemicals to which ephedrine, etc., belong, to alkanes, which is the transformation needed to turn it into meth or Dexedrine, comes up with a few interesting papers. See first of all *Journal of the Electrochemical Society*, April 1959, pages 325-27, by Wawzonek and *Journal of Organic Chemistry*, Volume 35, pages 1604-05 (1970), by Erickson. In these two papers, benzyl alcohols such as mandelonitrile and mandelic acid were reduced. They first converted the alcohol to an ester, and were able to reduce the former alcohol grouping to the alkane in a divided cell using mercury as the cathode. They found that it was absolutely necessary that a quaternary ammonium compound such as tetraethylammonium bromide, or tetramethylammonium iodide, or tetrabutyl ammonium iodide be present in the reaction mixture as electrolyte for the reduction to occur. Having other salts in the solution as current carrier didn't work. I considered tweaking this procedure, trying the waterbed water conditioner ingredient alkyl dimethyl benzylammonium chloride, to see if this quaternary ammonium salt would also work in the process. After some consideration, I decided against it because of the mercury cathode. My feeling is that mercury metal and less than highly skilled dope cookers don't mix. This procedure may very well work, so if someone feels they are practiced enough to handle and keep mercury, feel free to give it a go.

Another interesting article is to be found in *Tetrahedron Letters*, No. 25, pages 2157-60 (1979). In this paper, a wide variety of alcohols were reduced to the alkane with uniformly good results. Here too, they didn't directly reduce the alcohol. First they converted the alcohol to the methane sulfonic acid ester. Then they did the electric reduction in a divided cell using dry dimethylformamide as the solvent, and the quaternary ammonium compound tetraethyl ammonium p-toluenesulfonate as the electrolyte. The electrolyte allows current to

pass through the solution, as pure dimethyl formamide isn't a conductor. This is a good method, but isn't clandestine suitable. Dry dimethylformamide and the exotic quaternary ammonium compound aren't run of the mill chemicals. I also think this method would give off powerful odors if the methane sulfonic acid were to be reduced to methyl mercaptan. People would think you are raising skunks.

A much better line of pursuit is to be found in *Transactions of the Faraday Society*, Volume 60, pages 913-18 (1964) and Volume 55, pages 1400-20 (1959), *Journal of Electroanalytical Chemistry*, Volume 114, pages 273-81 (1980) and *Electrochimica Acta*, Vol. 21, pp. 449-50 (1976). In these articles, they found that if a metal which is a hydrogenation catalyst, such as palladium and platinum, is used as a cathode, i.e., it is connected to the negative terminal of a DC transformer, then the hydrogen generated at the surface of the catalytic cathode can be used to do convenient tabletop catalytic hydrogenations. This principle is also put to use in the next chapter where a Raney nickel cathode is the key to a convenient tabletop MDA synthesis. The unanimous finding is that the freshly generated atomic hydrogen at the catalytic cathode is a surprisingly powerful and effective means of doing a catalytic hydrogenation. The authors cited above wasted their time hydrogenating things such as ethylene. We can think of better things to hydrogenate electrically than that.

A series of related methods are known for converting ephedrine, pseudoephedrine or phenylpropanolamine (PPA) to meth or dexedrine respectively using catalytic hydrogenation. These are all good methods, but they haven't been able to approach the method using hydroiodic acid and red phosphorus in popularity among clandestine cookers. For complete coverage and comparison of these methods see Chapter Fifteen in *Secrets of Methamphetamine Manufacture*, Fourth Edition.

The explanation of why the HI and red P method has remained preeminent is probably twofold. All of the hydrogenation methods use palladium black as the catalyst. Even though this substance isn't on reporting lists to the feds, its only common source is from those dens of snitches at mainstream suppliers. Palladium black is easily made from palladium chloride. See *Secrets of Methamphetamine Manufacture*, Fourth Edition, Chapter Fifteen for details, or go to the *Organic Syntheses* collective volumes for the procedures. Even so, it would appear that getting palladium chloride from those snitches isn't much smarter. Note that it was disclosed in the Fourth Edition of the meth book that $PdCl_2$ is also sold by companies supplying precious metal salts to electroplaters of these metals. All of this speculation is now just academic anyway, because in this book a most simple and effective procedure is given for making your own $PdCl_2$ from an ingot of palladium, some hydrochloric acid, a couple of rubbers, and a source of DC current. I've tried this procedure, and the results are quite satisfying. Take it from your Uncle, no home should be without an ingot of palladium. Head on down to your local precious metals and coins dealer. Spend the $240 or more for one ounce of palladium. It'll be the best buy of your life.

The second reason why the HI and red P method has remained most popular is no doubt the lack of cheap, reliable and easily available hydrogenation equipment. See "The Poor Man's Hydrogenation Device," Chapter Thirteen in this book. An aluminum fire extinguisher is easily converted into a really good hydrogenation device that allows for magnetic stirring of the contents. Of course, the aluminum won't stand up to the sulfuric, acetic, hydrochloric, or perchloric acids used in these hydrogenation procedures, so the inside of the extinguisher should be coated with Teflon or Teflon loaded paint such as Xylan 1006. See *Vestbusters* for details on ordering and applying these useful materials.

But I digress. So which of the catalytic hydrogenation methods could be converted to an electrocatalytic process? Going to Chapter Fifteen of my meth book, the first procedure given uses dry HCl in acetic acid and palladium black to reduce ephedrine, or whatever, to the amphetamine. This procedure wasn't even considered by me for further research. The next procedure given requires that one first take the ephedrine hydrochloride, or whatever, that one has isolated from OTC pills, and add it to sulfuric acid to form the sulfuric acid ester. Electrocatalytic hydrogenation of this sulfuric acid ester in water solution proved to be a failure, giving back what I assume to be unreduced ester. Perhaps in alcohol solution it may work, so serious experimenters take note.

The next method given uses a mixture of sulfuric acid in acetic acid and palladium black to yield the amphetamine. Electrocatalytic hydrogenation of this mixture results in just a small raising of the octane numbers of the ephedrine fed into the process. The solution is just too strong to allow for a good rate of generation of hydrogen on the palladium cathode.

What does work is now given. First one starts with the OTC pills containing the ephedrine, pseudoephedrine or PPA, and extracts out the active ingredient using water as described in Chapter Fifteen of my meth book. Note that pills have become even more loaded with fillers since the publication of that book, so even more water is required to get complete extraction of the active ingredient from the pills. Then after adding a dash of HCl to the water extract and boiling it down to concentrate it, the water extract is made strongly alkaline with lye, the ephedrine extracted with a couple portions of toluene, and the toluene extract bubbled with dry HCl to give about 2 grams of crystalline ephedrine hydrochloride from each 100 lot of 25 mg ephedrine pills extracted. See the meth book Chapter Fifteen for a more complete discussion of this extraction and isolation procedure. Note that some brands of pills require a preliminary

soaking with toluene to degum and desplooge them prior to water extraction. Without doing the desplooge, these certain brands of pills will give a milkshake like emulsion upon being made strongly alkaline and extracted with toluene.

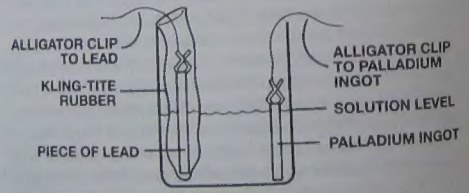
Now one gram of ephedrine, pseudoephedrine or PPA hydrochloride is put into a large test tube, along with 5-7 ml of glacial acetic acid. The bottom of the test tube is placed into a pot of hot water, and when the ephedrine hydrochloride, or whatever, is about all dissolved, a few drops of concentrated sulfuric acid are added. Mix it all in, and loosely stopper the end of the test tube with a cork to prevent steam from entering. Heat the hot water bath to just about boiling, and use this hot water bath to heat the test tube and its contents for a few hours. This forms the acetic acid ester of the ephedrine, pseudoephedrine or PPA, used in the reaction.

The solution should appear clear and water-like, and completely homogenous. After heating, the reaction mixture can be kept stoppered as is with no harm for at least a few days, but it's best to use it immediately after it's cooked and cooled.

This reaction to form the acetic acid ester is a typical ester forming reaction, and the usual rules apply. Water must be kept out of the reaction mixture, as its presence greatly reduces the yield. As a consequence, only crystalline ephedrine, pseudoephedrine or PPA hydrochloride can be fed into the process. A concentrated water extract won't do. An excess of acetic acid pushes the equilibrium toward making more ester. As a result, 7 or more ml of acetic acid is preferable to 5 ml. Only glacial acetic acid can be used, as diluted acetic acid is full of water. It would be best to reflux the ester forming mixture, but the simple procedure given here, heating to about the boiling point of water with precaution to keep steam from getting into the test tube, works good enough to give satisfying results.

Next mix up a solution of 5 ml of concentrated sulfuric acid in 100 ml of water. Take a 250 ml beaker, place it on a mag-

netic stirrer. Clip a well scrubbed Kling-Tite Naturalamb rubber in one side of the beaker, and put a piece of lead about ½-inch in diameter, and a few inches long, inside the rubber. On the opposite side of the beaker, stand up a one ounce ingot of palladium. Using alligator clips, make contact with the ingot, and with the piece of lead. Next pour most of the dilute sulfuric acid solution into the beaker. Save enough that some can be poured into the rubber so that the solution levels are about equal inside the rubber and the beaker at large. The ingot of palladium should be almost completely immersed. The alligator clip should be up out of the solution and there should be enough space left to add the ester reaction mixture from the test tube to the beaker without causing the solution level to reach the alligator clip. See the drawing below.



The choice of a 250 ml beaker here is based solely upon having room inside the beaker to put a standard size magnetic stir bar along with the two electrodes and rubber. A 100 ml beaker would no doubt be superior, as the one ounce palladium ingot would be considerably larger in relation to the catholyte

volume in a 100 ml beaker. The ester of ephedrine, pseudoephedrine or PPA would also be considerably more concentrated in a 100 ml beaker, allowing for more efficient electric reduction. In a 250 ml beaker, the ester reaction mixture will be diluted over 10 times by the catholyte whereas in a 100 ml beaker, the dilution will be more like five times. A perfectly usable magnetic stir bar can be made by cutting a section of bar magnet and coating it with a few coats of tough paint. In this way, a suitable size stir bar for the smaller beaker can easily be made.

A glass beaker isn't the only reaction vessel which can be used. The only requirements are that it be non-conductive so that the cell doesn't short out, and it must also be inert to the dilute acetic and sulfuric acid used in the process. A measuring cup with a pour lip would be a quite good substitute, and a drink tumbler would also be serviceable.

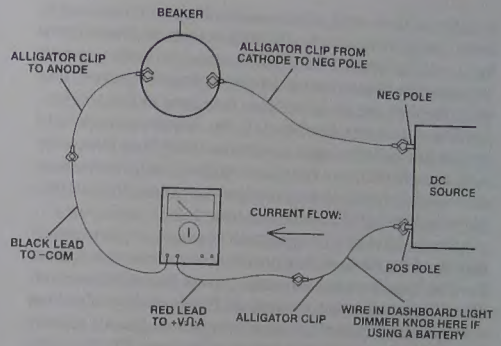
The lead anode shown in the drawing can be replaced with other materials as well. The only function of the anode is to pump current into the solution. It doesn't take part in the reaction in any other way. Suitable replacements for lead would be graphite rod obtained at the welding supply shop or dissected out of a dry cell battery. One could also use platinum metal. Unsuitable choices for anode materials include iron and steel, copper and brass, and aluminum. All of these metals will dissolve in dilute sulfuric acid when made anodic. The exact size or shape of the anode isn't particularly important. So long as it has dimensions about that shown in the drawing, it will work fine. As stated earlier, its only function is to pump current into the solution.

The rubber serves two purposes. It first of all keeps the ephedrine, pseudoephedrine or PPA from coming into contact with the anode. These substances will oxidize at the anode, resulting in cleavage producing benzaldehyde, methylamine or acetaldehyde. This makes one wonder whether the process

could be run backwards with electric reduction. Methamphetamine will oxidize at the anode to form a tar which clings to the anode surface. See *Chem. Pharm. Bull.*, volume 25, pp. 1619-22 (1980) for more on this subject. The rubber also serves to keep the oxygen which is generated at the anode surface from leaking into the solution by the cathode. This would interfere with the hydrogenation taking place there.

The surface of the palladium ingot should be lightly sanded prior to use. This increases its surface area a little, and exposes fresh clean metal. The piece of lead should be scrubbed free of grease and dirt. The wire lead to the palladium ingot can be clipped to the side of the beaker with a clothes pin or paper clip, to prevent the ingot from falling over during the course of the reaction. A DC current meter (amp meter) should be put in line on the wiring. A perfectly good one can be had at Radio Shack for about $50. Note that the model I have was made in China, and the instructions for how to wire it in to measure current were wrong. You'll figure it out, I'm sure.

First the wires are hooked up so that the palladium ingot is connected to the positive pole of the DC transformer, and the piece of lead to the negative. The typical one ounce ingot will have a face with an area of about 6 square centimeters immersed in the solution, and about one square centimeter up out of the solution. Only count the area on the side facing the lead piece. The back side doesn't count because current can't reach it. With this typical size ingot apply about 2 amps for one or two minutes. Oxygen will bubble freely from the ingot, and hydrogen from the piece of lead. Blackening will be noted on the edges of the ingot, where the current is most intense, and a lighter discoloration on the flat face of the ingot. This pretreatment is called "anodizing." It has been found that anodizing increases the ability of the palladium ingot to absorb hydrogen when the wiring is turned around, and the ingot is made the cathode.



Next, redo the wiring so that the palladium ingot is attached to the negative pole of the DC transformer, and the piece of lead to the positive. Turn back on the juice, and for this typical size ingot, run between one and two amps of current for about 20 minutes. At first, the amount of hydrogen generated at the palladium ingot will appear small, because it absorbs the hydrogen so well. After about 5 minutes of current passage, the whole surface of the ingot will freely bubble off hydrogen.

An alternative to the use of sulfuric acid electrolyte is to use 2% HCl solution. In this case, the ingot is first hooked up as anode, and a current of one or two amps is applied for a minute or two. The surface layer of the ingot will dissolve as a reddish brown solution of palladium chloride. Then the palladium ingot is made the cathode and about 50 milliamps per square cm of face applied for about 10 to 20 minutes. Most of the dissolved $PdCl_2$ will electroplate onto the surface of the ingot. The surface treatment is called "palladized palladium." Now "anodize"

in dilute sulfuric acid solution as in the previous example. Next, return the ingot to the 2% HCl solution and charge up the ingot with hydrogen for about 20 minutes as in the previous example. The electrocatalytic hydrogenation of the acetic acid ester of ephedrine, or whatever, is then done in this solution, just like in the example which follows. A lead anode can't be used in this variation, as it would dissolve. Other than being more complicated, this variation is probably superior to using dilute sulfuric acid, as this acid tends to poison the catalytic property of the palladium surface over time.

After the 20 minute charging with hydrogen, begin magnetic stirring of the solution, then pour in the ester reaction mixture from the large test tube. Adjust the current flow to between 35-50 milliamps per square centimeter of the ingot face. If one has 6 square centimeters of the ingot facing the lead anode actually immersed in the solution, a current of between 200-300 milliamps is called for.

This will result in some gassing off of hydrogen from the edges of the ingot, but over the rest of the surface of the ingot the hydrogen formed will react before it bubbles off. The lead anode will form a brown layer of lead oxide, and not dissolve at all in the sulfuric acid solution. Some surface particles will be kicked off the lead when it's first charging, but they don't make it through the rubber. The lead anode can be replaced with a piece of platinum if one desires, but lead is a lot cheaper.

Keep an eye on the current meter, and make sure that the current flow stays in the 200-300 milliamp (.2-.3 amp) range for the size ingot given in this example. Allowing too much current to flow will cause the ingot surface to be covered by hydrogen bubbles, and the solution won't be able to come into free contact with the metal surface. Turning the current down too low may result in no hydrogen being formed at the palladium surface.

The best and most convenient DC electric power source is a rectifier such as those commonly used by electroplaters to do lab scale electroplating testing and experimentation. Such rectifiers cost about $500-600 from suppliers of electroplating equipment. Using such a device, the current flow is easily controlled by turning up or down the voltage output of the rectifier. The higher the voltage output, the more current is passed through the solution. E=IR.

The next best current source is a 12 volt car battery with its voltage output modulated by hooking into the wiring to the beaker a dashboard light control knob. This dashboard light control will cost a few bucks at the auto parts store, and will function in this electric cell just as on the dashboard. Turn the knob up, as you would to brighten the dashboard light, and the voltage is increased and more current will pass through the solution.

A toy train transformer may also work, but beware of a thing called "AC ripple" found with such cheap power sources. This is where AC current is superimposed upon the DC current. Generally it will produce a "picket fence" output visible on an oscilloscope. So long as the spikes all run in the desired direction, I think that it will work OK. If, on the other hand, the AC ripple causes the palladium ingot to oscillate between anodic and cathodic, you're in for trouble.

When about 3000 coulombs have passed through the solution, the process can be considered to be complete for a one gram batch. A coulomb is one amp-second, so let's use the 300 milliamp current flow to calculate reaction time. 3000 amp-seconds divided by .3 amp = 10,000 seconds, or 2 hours 45 minutes.

Three thousand coulombs per gram of feed material has been found to give good yields of a fine product, but by no means assume this number to be the optimal. It may well be that greater yields would be obtained by passing more current. It

may also be that pseudoephedrine and PPA differ from ephedrine in their ease of electrocatalytic hydrogenation, and again require the passage of more current. I don't think that any harm can be done by passing too much current, within reasonable limits, so by all means experiment with the amount of current passed.

During the course of the reduction, the color of the reaction mixture slowly changes from its initially clear color to slightly tinted with yellow. It's not known if this color change is due to some of the Kling-Tite rubber soaking out to form a tea, or if it is the result of the reaction. In any case, this is a remarkably clean reaction.

When the desired amount of current has passed, the work-up and isolation of the product is very simple. The Kling-Tite rubber is removed from the beaker. After pulling out the lead or platinum anode, the rubber is flushed down the toilet. The anode can be reused over and over. Then the palladium cathode is removed, and rinsed off. It too can be reused an innumerable number of times. The process of "anodizing" the palladium will have to be repeated prior to each run. Some fresh metal may have to be exposed on occasion by light sanding of the metal surface. An ingot of palladium should last for a lifetime.

The reaction mixture should be poured into a sep funnel, and approximately 20% solution of NaOH (lye) in water should be added with shaking until the mixture is strongly (13+) alkaline to pH paper. Then extract with one or two portions of toluene. Fifty to one hundred ml of toluene is more than enough to extract one gram of product. The toluene extracts are next bubbled with dry HCl to get the crystalline hydrochloride product. After filtering and rinsing them off with some fresh toluene, they are spread out to dry. The most pleasantly surprising finding is that crank produced by this method doesn't give one the body and soul wrenching hangovers so typical of the product made by the HI and red phosphorus method. This

process is a highly desirable way to keep one's own party rocking and rolling.

If one should wish to produce more than a gram or so at a time, a larger palladium catalytic cathode should be used. Linking together more ingots of palladium would get pretty expensive, so a more economical alternative will be detailed. That alternative is electroplating some copper or brass screen with a thick coating of palladium.

The simplest way to get this section of screen electroplated with palladium is to go to the yellow pages, look under electroplaters, and find one who electroplates palladium. Ask for a plate build-up of several thousandths of an inch thickness, so that enough palladium is deposited to last for a while.

This palladium plated screen would then be used exactly like an ingot of palladium. First it must be "anodized," then charged up with hydrogen in exactly the same way. The sole difference is that the greater surface area of the screen facing the rubber ensconced anode requires a correspondingly greater amount of current be passed. Then during the course of the reduction, again 50 milliamps per square centimeter of surface area facing the anode is used. The total of 3000 or so coulombs per gram of feed material doesn't change by increasing the size of the catalytic cathode.

An alternative to sending a section of screen out to be plated is to plate it yourself. One starts with an ingot of palladium, and anodically dissolves a portion of it to form a $PdCl_2$ solution. Follow the directions for doing this in the $PdCl_2$ chapter in this book. The procedure given here using a Kling-Tite rubber as a shield for the negative pole of the circuit works very well in my experience. The concentration of $PdCl_2$ in solution is found by weighing the palladium anode as it dissolves. The amount dissolved times 1.7 is the amount of $PdCl_2$ in solution.

A bath for electroplating palladium can be made up as follows:

palladium chloride	30-50 g/liter
ammonium chloride	30 g/liter
hydrochloric acid	adjust pH to .1-.5

With this plating bath made up, the section of copper or brass screen can be plated. First scrub off all dirt and grease with hot dishwater. Make sure there is no lacquer on the screen. Then rinse well, and immerse the screen in 5% HCl for about 30 seconds. The copper will take on a more intense red color as the surface oxides are removed in the acid. Rinse thoroughly, and if it is going to take more than a few seconds to get the screen into the plating bath, leave it immersed in rinse water.

Now the plating bath is put into a beaker, and an ingot of palladium (the remnants of $PdCl_2$ production will serve well) is partially immersed into the plating bath. I say partially, because the alligator clip connection shouldn't come into contact with the plating bath. The palladium ingot is attached to the positive pole of a DC transformer. Now take the section of screen, and attach it to the negative pole of the DC transformer. Make sure it stays wet from the rinse during this manipulation, so that the surface doesn't go passive, causing bad adhesion of the electroplate.

Turn on the DC transformer, and apply a few volts. Then immerse the section of screen. Make sure that the screen doesn't contact the Pd ingot, as this would cause a short. Once immersed in the plating solution, adjust the current flow to .03 amp per square inch of screen facing the palladium anode. Begin stirring the solution once the screen is in the solution. Plate the section of screen at the specified current rate for about an hour. When done, remove it and rinse it off. It's ready to use. The plating bath will keep forever in a tightly stoppered

dark bottle. It can be reused to plate new catalytic cathodes whenever desired.

Note: It is no doubt a good idea to flame the surface of the palladium ingot or electroplate for about 15 seconds with a propane torch after each batch is done and the ingot well rinsed. This flame treatment will burn organics out of the metal surface that may cause the surface to lose catalytic activity. It will also destroy evidence traces on the ingot. Regular flame treatments should greatly extend the useful life of the metal surface, and delay the need for sanding the surface.

Definitions

Anode: the electrode in an electrochemical cell which is attached to the positive pole of a DC transformer or rectifier. Oxidations occur here.

Cathode: the electrode in an electrochemical cell which is attached to the negative pole of a DC transformer or rectifier. Reductions occur here.

Divided Cell: a method of constructing an electrochemical cell wherein the solution in contact with the anode is kept separate from the solution in contact with the cathode. A cell divider is used to keep the two solutions separate. It must pass current, and be unaffected by the solutions used. Rubbers are the cell dividers most often used in this book.

Electrolyte: a material added to the solutions in an electric cell which allows the solutions to carry current. Pure water or other solvents just aren't conductors. Dilute acids or bases make good electrolytes in water, as do salts.

Anolyte: the solution in a divided cell which fills that portion of a divided cell in contact with the anode.

Catholyte: the solution in a divided cell which fills that portion of a divided cell in contact with the cathode. The anolyte and

catholyte are often pretty much identical with the exception of the substrate or precursor being acted upon.

Chapter Three

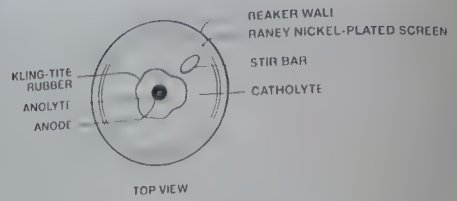
A Convenient Tabletop MDA Synthesis

There has long been a need for some simple and efficient method of making MDA and other amphetamines from their phenylacetones. The long accepted best method is catalytic hydrogenation of the phenylacetone with either ammonia, or better still, ammonium acetate, which serves as both ammonia source and condensing agent to encourage the formation and stability of the Schiff's base.

The problem with this procedure is that it uses Raney nickel as the catalyst for the hydrogenation. Not only is this catalyst pretty much exclusively available from those snitch filled scientific supply houses, it further is cursed by being quite similar to iron filings in its attraction to magnets. This magnetic property means that this catalyst is unsuitable for use in simple makeshift hydrogenation equipment such as "The Poor Man's Hydrogenation Device" found in this book.

An alternative to catalytic hydrogenation using Raney nickel is given in Dr. Shulgin's book *PIHKAL*. In that book several examples are given of reducing a mixture of the phenylacetone and ammonium acetate in methanol solution with sodium cyanoborohydride. See the MDA recipe in that book for a typical procedure and result.

The disadvantages of this alternative procedure are the relatively complex cleanup problems of supply of the electrolyte, the method just plain doesn't work very well in practice, and the rather high, with a reaction time of several hours at mild reflux. With a reaction time of several hours at mild reflux, with a reaction time of several hours at mild reflux, with a reaction time of several hours at mild reflux, with



To construct the cell, one starts with a section of brass or copper screen cut so that its height will be short enough that all of the screen will be submerged by the hydrogenation mixture, and long enough that the screen will line the inside surface of the beaker used in the electrocatalytic hydrogenation. The screen must be entirely submerged because Raney nickel will ignite if it dries out. The hydrogenation mixture is flammable, so this is a bad combination. One can always add more methanol to the mixture if you are a little short on batch size.

The anode is most preferably a rod of graphite about ½ inch diameter obtained from the welding supply shop nearest you. Platinized mesh would also work, but it costs plenty. The anode should be in a central position relative to the Raney nickel cathode. The placement of the anode is important. If it is offset from center, the current density on the Raney nickel cathode will be greater where the anode is closer, and less where it is more distant.

To make a Raney nickel plated screen, one starts with that section of brass or copper screen described above. Don't use aluminum screen, as this metal is difficult to plate. Ditto for stainless steel screen. You have to be a plater who knows this artform to use these screens. Copper and brass are easy,

quite suitable for beginners to electroplating. The screen should be new and clean to begin with, but first scrub the section of screen with hot water using dish soap. Rinse it off thoroughly with hot water, then sit the screen on paper toweling to keep it clean. Have clean hands to avoid smearing grease on the screen.

Next thread a copper wire through the screen near its top edge, and twist it down tightly to assure good electrical contact. Then immerse the copper or brass screen in 5% hydrochloric acid solution for about 30 seconds or so. This removes oxides from the surface of the copper or brass, and leaves the surface active and ready to accept an adherent electroplate. Rinse the screen in clean water. You will note that the screen is now more reddish colored.

Immediately after the water rinse, and while the copper or brass screen is still quite wet with the rinse water, immerse the screen into a plating solution containing the following ingredients in water solution: $NiSO_4$ $6H_2O$ (120 g/liter); NH_4Cl (15g/liter); H_3BO_3 (15g/liter). The nickel sulfate can be replaced with nickel chloride if this chemical is more handy. The boric acid additive is quite important and shouldn't be left out.

Most importantly of all, add about 10 grams of unleached Raney nickel alloy (50% nickel, 50% aluminum) for each 100 ml of plating solution. The Raney nickel alloy should be 200-325 mesh. This particle size corresponds to a fine grit. My experience with melting Raney nickel alloy and grinding off a fine grit is given later in this chapter. Begin stirring to keep the particles suspended in solution.

The best container for doing this electroplating is a beaker about the same size as the anticipated batch reduction container. It could even be the same beaker. Put the section of screen along the inside wall of the beaker, just as shown in the figure given earlier in this chapter for the reduction cell.

Run the copper wire to the negative pole of a DC transformer. In the center of the beaker put a piece of nickel, iron or graphite rod as the anode. Attach it to the positive pole of a DC transformer. Apply a current of .07-.1 amp per square centimeter of the screen surface facing the anode. Don't count the surface area on the back side. Plate with stirring for about 15 minutes. No rubber needs to be used in this electroplating operation. If a nickel anode is used, the nickel will dissolve at a rate that matches the rate that it plates out at the screen. This will assure that the plating solution can be used many times without problems. Iron anodes will contaminate the solution and ultimately the plate with iron, but that shouldn't be harmful at least in the short run. The only purpose served by the nickel plate is to be sort of a metallic glue to paste particles of Raney nickel alloy to the screen.

The plating solution and the necessary wiring, including amp meter, should be set up and ready to go prior to beginning the acid dip of the copper or brass screen. Undue delay in beginning the electroplating of the screen after the acid dip and rinse can result in the surface of the copper or brass screen becoming passive and unable to accept a strongly bonded electroplate. This will result in flaking and peeling of the plate. After the 15 minutes of electroplating, the screen should be entirely coated with a dull gray colored nickel plate liberally studded with particles of Raney nickel alloy. They should be obvious to see, and take up about 20% of the surface area of the screen.

At the end of the electroplating process, turn off the current, remove the screen from the plating bath, and give it a water rinse. After air drying the screen, it can be kept indefinitely in a dry place.

The phenylacetones used in this procedure are best made from essential oils containing the desired carbon skeleton as

a major ingredient. For a thorough discussion of this topic, see the Second Edition of *Practical LSD Manufacture*. As an example, calamus oil will generally contain a large percentage of asarone or B-asarone. This substance has the carbon skeleton of TMA-2. On the side chain, it has a double bond in the propenyl position. The phenylacetone can be made by the general procedure given in *PIHKAL* using formic acid and 30% peroxide to form the glycol, and then when the glycol is heated with dilute sulfuric acid, the phenylacetone is formed. A better procedure is given in *Practical LSD Manufacture*. In an undivided electric cell with a central graphite anode surrounded at a distance of 1 cm with stainless steel cathode, the epoxide is formed. Rearrangement of the epoxide gives the phenylacetone. Also see *Journal of Organic Chemistry*, Vol 46, pp. 3312-3315 (.1981), and *Journal of Organic Chemistry*, Vol. 49, pp. 1830-1832 (1984).

When the essential oil contains the desired carbon skeleton with the double bond in the allyl position, as for instance safrole in sassafras oil, then the best way to make the phenylacetone is by the so called "Wacker Oxidation."

Enough background, this chapter is about how to use a Raney nickel cathode! The section of screen which has been plated with a composite of nickel and Raney alloy is immersed in 300 ml of distilled water. Then add 50 grams of sodium hydroxide pellets, and stir them into solution. An exothermic reaction occurs with a lot of fizzing off of hydrogen as the aluminum dissolves out of the Raney alloy. When the fizzing slows down, heat the mixture to 60° C for 30 minutes with occasional stirring of the solution. Then let the mixture cool to room temperature.

When the mixture has cooled down, pour out the sodium hydroxide solution, and begin a thorough rinsing of the

screen with water. Hydroxide clings to surfaces, so at least four swirling rinses of the screen will be required.

Immediately after the rinse, place the Raney cathode screen into the electric cell diagrammed earlier in this chapter. For illustration, consider a batch using 30 ml of methylenedioxyphenylacetone made from safrole. A 250 ml beaker would be about the right size container in which to do this batch. A screen about 3 cm high, and 20 cm long would nicely line the lower inside wall of the beaker. A stock solution containing 12 grams of ammonium acetate, 1.8 ml of acetic acid, and 3 ml water in 100 ml of methanol is made up, and the majority of it is added to the beaker. Enough is poured into the rubber to equalize the liquid depth inside the beaker and the rubber. One must get this solution into the beaker before the screen dries out, because dried Raney nickel will start fires. The screen must be completely submerged by the solution.

Next, connect the screen to the negative pole of a DC transformer. The anode should be connected to the positive pole of the transformer, and an amp meter should be put in line on the wiring. The wiring which serves the anode is the correct place to place the amp meter. Begin a flow of about 5 amps through the cell. This is a current density of about 50 milliamps per square centimeter of the screen face. A gentle evolution of hydrogen will be noted from the screen. Continue passing current at this rate for about ½ hour to charge the Raney nickel with hydrogen fully.

Now add the phenylacetone to the beaker, and begin stirring. Turn down the current flow in the cell to between 1 and 1.5 amps. This corresponds to 10-15 milliamps per square centimeter of the screen face. About 3 faradays per mole of phenylacetone should pass through the cell. The methylenedioxyphenylacetone has a molecular weight of 174 and a density of about 1, so 30 ml is about .17 mole. A

faraday is 96,500 coulombs or amp seconds, so 3 faradays times .17 mole equals about ½ faraday, or about 50,000 coulombs. At a current of one amp, this takes 13¾ hours.

When the required amount of current has passed, turn off the transformer or rectifier, and remove the rubber and its anode from the beaker. Then lift the Raney nickel cathode from solution, and rinse it with alcohol. Store it under alcohol. It should be good for reuse within the next couple of months.

Then pour the reaction mixture into a vacuum flask or similar vacuum container, and evaporate away most of the alcohol under a vacuum. Warm water and shaking the contents speed the evaporation. Pour the residue after the evaporation into a sep funnel, and add 20% sodium hydroxide solution in portions, with shaking between adds of hydroxide. Cool the mixture in water if it gets too hot. Continue adding hydroxide until a product layer forms on top of the water layer. Check the pH of the water layer. It should be 13+ to pH paper. If not, add more hydroxide with strong shaking between adds.

Next extract out the product with about 100 ml of toluene. Separate the toluene-product layer. It should be no darker than yellow. It should also reek of ammonia. Remove the ammonia by some vacuum evaporation in a vacuum flask. It will be obvious to the nose when it is gone. Then dilute with more toluene until a total solution volume of about 250 ml is reached. Next bubble the solution with dry HCl to produce crystals of the substituted amphetamine hydrochloride product. They are filtered out using a vacuum assist with a Buchner funnel as the toluene solution of the product thickens during the course of the HCl bubbling. The filtered crystals are rinsed with fresh toluene, and spread out to dry. This process is discussed in detail in Chapter Five of *Secrets*

of Methamphetamine Manufacture. The total yield of crystals of MDA hydrochloride should be around an ounce.

There is a possibility that an oil layer may form and settle to the bottom of the beaker during the HCl bubbling. This is most likely due to the presence of a lot of unreacted phenylacetone in the toluene extract. This would be caused by poorly prepared Raney nickel, or a plated screen that didn't have many particles of the Raney alloy imbedded in it. If this happens, shake the oily toluene with an equal volume of 10% HCl solution for about 5 minutes. The product will go into the water solution, while the phenylacetone will remain in the toluene. Keep the toluene layer for recovery of the phenylacetone. Make the water layer strongly basic (pH13+) with sodium hydroxide solution, and extract with toluene.

The toluene layer should be separated, and bubbled with dry HCl gas to get clean crystals of MDA hydrochloride.

Melting Raney Nickel Alloy

To melt an approximately 20 gram batch of Raney nickel alloy, one should start with a crucible at least 20-25 ml in size. The high alumina crucible made by Coors is preferable to regular porcelain crucibles, because the high alumina crucible has a maximum rated temperature limit of 1750° C, versus 1300° C for the porcelain crucible.

Suspend this crucible on a ringstand. If one has a ceramic triangular shaped crucible holder, this is the preferred tool for the job. Lacking this item, one can take stainless steel rod about 1/8 inch diameter and bend it around some pipe of the same diameter as the crucible. This stainless steel rod will make a good crucible holder, so long as it rides well up the sides of the crucible where the heat isn't so intense. Two of

these stainless steel holding rods placed at right angles on the crucible will give good stability.

Next dress up in your welding outfit. This includes a clear face shield, a little beaney to cover the top of your head, leather jacket and leather gloves. Whether your genitals deserve protection is up to you. A fan should also be set up to carry metal fumes downwind. I didn't notice much fuming in my experiments.

Then take 10 grams of aluminum foil, and fold it over on top of itself repeatedly to get a compact bundle. Pack it down tightly, then cut it in half. This will be about all you will be able to stuff into a 20 ml crucible for starters. Place an oxy-acetylene torch under the crucible, and light its fire. Don't turn up the gas flow rate to a level suitable for metal cutting, just have a nice blue flame under the crucible and heat its bottom. A simple propane torch would also be hot enough to melt this alloy, as an air-propane flame has a temperature of 1900° C, compared to 2300° C for air-acetylene, and still higher for oxy-acetylene. Too aggressive heating with an oxy-acetylene torch will burn through the crucible. A hotter burning alternative to propane (MAPP) may also be too hot.

The aluminum will soon start to melt, making room for the rest of the aluminum foil in the crucible. Aluminum melts at 660° C, quickly forming a puddle of liquid aluminum in the bottom of the crucible. When the aluminum has mostly all melted, add 10 grams of nickel metal cut up into approximately split pea size. Large wire cutters will serve to cut up pieces of nickel metal. Make sure the nickel pieces are clean before putting them into the pot. There are several sources of pure nickel metal. I'm told that old nickels actually were made of nickel. Lyndon Johnson debased the coinage in the mid-60s, and now they may be nickel plated junk. Pure nickel can be obtained by going to an electroplater who plates nickel, and asking to buy a few of his nickel

anodes. They come in pieces about the size and appearance of chicken turds, and are of quite high purity. Tell him you plated them into jewelry or some such story, and you should have no problem talking him out of a few of them. One could also ask for a nickel Hull Cell anode. This is a rectangular piece of nickel about 2½ inches square and about 1/8 inch thick, and will sell for under $10.

Pure nickel metal melts at 1555° C, but the melting temperature will be lower than this in the pot with molten aluminum by an effect similar to the lowered melting temperature of ice in alcohol. The melting of nickel into molten aluminum to form an alloy is also a quite exothermic process, so if using an oxy-acetylene torch, take care with the heating.

Once the pieces of nickel have been in the pot for a minute or so, some stirring is called for. If one has a nickel Hull Cell anode, cutting a thin strip off the side of the anode makes a good stirrer. Hold it with a pliers, and stir the molten metal around for a few seconds, then remove the stir stick from the pot and let the stick cool off. Repeat the stirring every minute or so until the pieces have all melted, then continue for at least a few minutes longer to assure a homogenous alloy. A rod of stainless steel can also be used as a stir stick, but it's resistance near as good as a nickel metal stick. Be careful the steel doesn't start to melt on the first stir. The remelting of the alloy which clings to the end of the stick will help to prevent contamination on succeeding stirs.

When a homogeneous alloy has been achieved, stop the heating, clean up the crucible and let it cool. If the inside surface of the crucible isn't too pitted, the chunk of alloy will fall right when tipped over and tapped on a book cover. A pitted crucible may have to be broken to remove the alloy. Freezing in a freezer to contract the alloy before resorting to hammer

The homemade Raney alloy will be grey colored, and a bit of it will be pulverizable using the hammer. So beat on the chunk of alloy, gently, being sure not to let fragments escape. The residue which isn't pulverizable with a hammer should be placed on a piece of paper, held in place with a vise grip or pliers and filed on with a fine toothed file to reduce this remnant to a fine grit. The powdered alloy and the filed grits should be placed in a clean glass jar or baggie until used.

EFFECTS OF SOME VARIABLES UPON THE AMOUNT OF RANEY NICKEL ELECTROPLATED UPON THE CATHODE

TABLE I. Effect of Particle Powder Concentration on the Quantity of Ni Powder Produced in the Cathode and Cell Voltage Efficiency. (Plating at 1 amp/dm²)

Concentration of Ni Powder in Electrolyte g/l	Quantity of Powder Produced g	Cathode Efficiency %
0.5	5.00	100.0
1.0	9.98	99.8
1.5	14.9	99.3
2.0	19.8	98.9
2.5	24.7	98.8
3.0	29.6	98.7
3.5	34.5	98.6

3 Amps/ dm² (1.5 gal. 1.0 amp powder

TABLE II. Effect of Cathode Current Density on the Volume Percent and Cathode Efficiency

Cathode Current Density Amps/dm²	Volume % Deposit %	Cathode Efficiency %
1.0	20.8	97.5
2.0	42.7	99.7
3.0	64.6	100.0
4.0	86.5	100.0

Particle concentration 2.5 g/l

TABLE III. Effect of Solution Temperature on the Volume Percent and Cathode Efficiency

Temperature °C	Volume % Deposit %	Cathode Efficiency %
20	42.7	99.7
40	42.8	99.9
60	42.9	100.0

TABLE IV. Effect of Solution pH on the Volume Percent and Cathode Efficiency

pH	Volume % Deposit %	Cathode Efficiency %
2.5	42.4	99.0
3.5	42.7	99.7
4.2	42.9	100.0

rubbers can be used similarly, but they have ...ted difficulties. The biggest problem with latex ...ers is that when they are new, they have a pretty ...stantial electric resistance. Applying over 20 volts to a ...and new rubber will still not cause it to pass current. I can't tell you how many volts are required to break the insulation on a new rubber, because 20 volts is all I can get out of my DC transformer. As rubbers made of latex age, however, their resistance lowers markedly. My house is just full of old rubbers, and I have found that Trojan Extra Sensitive condoms a year or two past their expiration date will pass current when about 5 volts is applied to them. These old rubbers work admirably to separate the anolyte from catholyte, and are also quite resistant to diffusion. Their ability to stand up to acids and bases is also quite good. If one has an old package of rubbers around the house, take one out, scrub it up, and see how well it passes current. There may very well be solutions in which the use of an old rubber is preferable to using a new Kling-Tite.

Chapter Five

Preparation Of Palladium Chloride

One of the most useful substances a serious cooker can have is palladium chloride. From making palladium black catalysts for hydrogenation, to the so called "wacker oxidation" wherein $PdCl_2$ catalyses the transformations of allylbenzenes such as safrole from sassafras oil to phenlyacetones, to your own palladium plating bath to plate "catalytic cathodes," palladium chloride plays a central role.

The last time I checked, $PdCl_2$ was still pretty easily available from outlets which supply precious-metal salts to electroplaters, but this situation is likely to change in the near future. As a consequence, a simple procedure for making one's own supply of this substance in a very low-profile manner is of obvious value.

To make palladium chloride, one starts with a glass bread loaf baking dish or similar glass container. One also needs a thoroughly cleaned condom made of rubber or sheep guts. The extra thin, sensitive brands are best as they have less resistance to passage of electricity.

The next thing one needs is an ingot of palladium. These can be obtained from one's local dealer in precious metals. (You're hedging against an outbreak of inflation or monetary

example). One troy ounce will cost upwards of $240, and make a couple of ounces of $PdCl_2$. If small ingots are available, they may be the way to go.

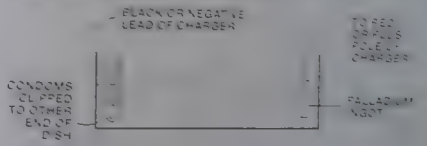
Take the ingot, and either hammer on a corner or squeeze a corner in a vise to make an elongated lip on which to make electrical contact. This will allow contact to be made to the ingot without immersing the copper jaws from the battery charger in the hydrochloric acid solution, thereby contaminating it with copper. Palladium is, in general, fairly malleable, but if it has been cold-worked, its strength increases greatly. If you have trouble hammering or squeezing out a lip for contact, try heating the ingot to redness on a stove burner.

The next step is to paint the exposed copper on the red or positive lead from one's battery charger. Leave the teeth on the lead unpainted so that they can still make electrical contact. The paint prevents acid mist, which will come up from the solution, from dissolving copper into it.

Now pour hydrochloric acid into the baking dish or beaker until it's about half full. The exact strength of the hydrochloric acid isn't critical, but lab-strength (35%) HCl cut in half with water is a reasonable choice.

Place the palladium ingot into the HCl solution. The lip made for electrical contact should be up out of the solution so that copper from the charger's jaws doesn't get into the solution. Attach the red or positive charger lead to the palladium ingot.

On the opposite end of the dish, two condoms, preferably Trojan Extra Sensitive brand, are rolled one on top of the other to form a double thickness rubber barrier. The inner condom is filled with the same hydrochloric acid solution so that the liquid depth inside the condom about equals that in the dish. Some of the acid is also poured into the space between the two condoms so that current can flow. Insert the black or negative lead of the charger inside the condom, immersed in its supply of acid. See the illustration below. In my experiments with this process, I found that the double layering of the condoms was absolutely necessary, as a single condom was just too permeable to the hydrogen formed at the negative pole. If this hydrogen should get into the solution, it could sludge out the palladium chloride.



Begin magnetic stirring in the dish, and turn on the charger. First try the 6 volt setting, and check the current meter to see if any current is being passed. In all probability, no current will flow at 6 volts. Trojan Extra Thins don't pass current until 12 volts is applied, and thicker condoms will require even higher voltage to break the insulation. Adjust the voltage so that a slow current flows. For an ingot about an inch long and half inch wide, no more than an amp or two should flow. Smaller pieces of palladium should have a proportionally smaller amount of current applied. This must be DC current, not AC! A transformer may have to be put in line to adjust the current to the proper range. The black lead inside the condom will, of course, fizz crazily, producing hydrogen, but one wants to avoid production of oxygen or chlorine at the palladium anode. The sole anode reaction should be the dissolution of the palladium metal to make a solution of $PdCl_2$.

Failure to pass current is most likely due to insufficient voltage being applied to break the insulation of the condoms, but one should also check to make sure that a good contact is being made on the palladium ingot. This ingot must be well cleaned and grease-free prior to immersion in the acid.

At a current of one amp, 54 hours are required to dissolve 106 grams of palladium. One ounce will dissolve in a little over 14 hours. Some sludge may form from flaking off the palladium anode, but so long as the hydrogen made at the cathode doesn't get into the solution, sludge formation will be minimal.

When the palladium ingot has mostly dissolved, turn off the current. Pull the condoms out of the solution, and also remove the remnant of the palladium ingot. Weigh this remnant, then filter the hydrochloric acid solution containing the $PdCl_2$. The filtered out sludge should also be weighed when it's dry.

The combined weight of the remains of the ingot and the recovered metal sludge can be used to figure how much $PdCl_2$ is in the solution. The amount of palladium metal dissolved times 1.7 equals the weight of $PdCl_2$ in solution.

Next, the excess acid should be removed from the solution. This is done by boiling the solution. Distilled water should be added to the beaker while it is boiling to make up for that which boils away, maintaining the original solution level. When the steam coming off the beaker no longer gets a strongly acid reaction from pH paper, the process can be considered completed. The volume of the solution should be measured, and the amount of $PdCl_2$ per ml calculated. Then the solution should be bottled up, tightly stoppered, and stored in a cool dark place like the refrigerator. The water solution of $PdCl_2$ can then be used as a source of $PdCl_2$ and water.

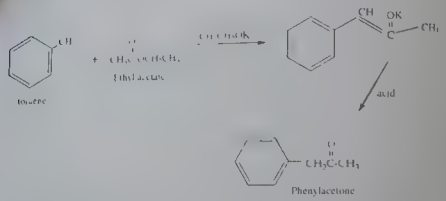
Chapter Six
A New Low Profile Phenylacetone Synthesis

I'm always in the mood for a new phenylacetone recipe, especially when it uses cheap, commonly available ingredients. I'm sure you are too. That's why when a correspondent named Chris sent me this procedure, I got a smile on my face and said, "This has to be published."

This method is based upon the procedure given in *Organic Syntheses,* Collective Volume 5, pp. 567-71. The name of the recipe is ethyl indole-2-carboxylate. As given, it's an interesting indole synthesis, and that's all. However, when one substitutes toluene for the nitrotoluene in the recipe, and ethyl acetate for the diethyl oxalate in the recipe, one gets phenylacetone rather than ethyl nitrophenylpyruvate in the first stage of the reaction scheme.

This procedure is similar to the phenylacetone recipe using benzyl cyanide as the starting material found in *Secrets of Methamphetamine Manufacture,* and also in *Organic Syntheses.* The key difference here which allows toluene to be used rather than that List I chemical benzyl cyanide is potassium metal. In contrast to sodium metal, potassium is active enough to cause a linkage with toluene directly. There's no need for a leaving group like cyanide to be

attached to the toluene when potassium metal is used. The reaction sequence is illustrated below:



To do the reaction, a 5000 ml three-necked round bottom flask is fitted with a dropping funnel, a motor driven stirrer with seal, and a reflux condenser protected with a calcium chloride drying tube. Put 300 ml of anhydrous ether in the flask, along with 39.1 grams of freshly cut potassium metal. Potassium metal is some nasty stuff, at least as active as sodium metal. Care must be taken to protect oneself. Gloves and eye protection are mandatory. The chunk of potassium metal should be immersed in xylene (found in the paint section of the hardware store) contained in a mortar. A beaker is just too fragile to use as a container when cutting up this chunk of potassium metal. A Tupperware container would probably also work. Check its plastic for resistance to xylene prior to use. Then while the chunk of metal remains immersed under the xylene, scrape off the discolored outer skin of the potassium metal to reveal the shiny metallic luster of good potassium. A clean, sharp steel blade is OK for cutting potassium. Toss the scraped off skin into another container holding some xylene, so the metal remains immersed.

When the skin has been removed, cut up the potassium into pea-sized pieces. Then quickly remove a piece with a tweezer, blot it really quick with some filter paper, and drop it into a beaker on a scale which contains enough xylene to keep the piece covered with liquid. Note the weight before and after adding the piece of potassium. The weight gain is the amount that chunk weighs. Toss that chunk into the reaction flask. Continue weighing and adding chunks until 39.1 grams have been added.

The remaining block of potassium metal should be kept under its protective liquid. The scrapings and residue of potassium on the knife are very dangerous, and likely to start a fire if not immediately decomposed. The little flakes floating on top of the xylene are especially dangerous. Move the beaker containing the scrapings well away from the ether into a well ventilated area. Have a cover ready for the beaker to extinguish any fire which may well start. With a medicine dropper, add 91% isopropyl alcohol (t-butyl alcohol is much better) one drop at a time to the xylene and potassium scrapings. No other flammables or source of flame should be nearby. Continue adding alcohol until all of the metal has dissolved. Then flush it down the toilet. Repeat this procedure with the mortar or Tupperware container used to cut the potassium metal.

Never touch potassium metal! All manipulations and transfers of this metal should be done using a tweezer or similar tool!

When all 39.1 grams of potassium are in the flask, and the residues are decomposed and flushed, and the block of potassium metal is returned to its container, the reaction can be done. Flush the flask with nitrogen to prevent the danger of fires, then with stirring add a mixture of 250 ml of absolute ethanol and 200 ml of anhydrous ether from the dropping funnel just fast enough to maintain mild boiling.

When all of the potassium metal has dissolved, the slow flow of nitrogen to the flask that was maintained after the initial flushing can be stopped. It takes about 1½ to 2 hours to dissolve the potassium with stirring. Up to 3 hours without stirring.

The solution is then allowed to cool to room temperature, and then 2500 ml of anhydrous ether is added, followed by 98 ml of ethyl acetate. After stirring this mixture for ten minutes, add 106 ml of toluene, and stir for an additional ten minutes.

Now transfer this solution into a 5000 ml Ehrlenmeyer flask, stopper it and let it sit in a cool place for at least 24 hours. A lumpy, crystalline mass of the potassium salt of the enol of phenylacetone should separate out. Collect these crystals by filtration. Crystals clinging to the sides of the Ehrlenmeyer flask can be loosened and scraped out with a glass rod. After the crystals have air dried a bit, they should be added to 200 ml of glacial acetic acid. After some swirling, a yellow, opaque solution of phenylacetone should form. Next, slowly, with stirring or shaking, pour this solution into 1000 ml of water containing 130 grams of sodium hydroxide (lye). This neutralizes the acetic acid to form sodium acetate. The phenylacetone should float on top. Extract the solution with two 100 ml portions of toluene. The combined toluene extracts, which contain the phenylacetone, should be washed by shaking with about 500 ml of 5% NaOH solution, then the top layer, which contains the phenylacetone dissolved in toluene, should be transferred to a distilling flask.

Fractional distillation is commenced, just as described in *Secrets of Methamphetamine Manufacture*, Chapter Three. First some toluene-water azeotrope distills at 85° C. It has a milky appearance. Then pure toluene distills at 110° C. When most of the toluene has distilled out, heating is

stopped, and a vacuum is applied from an aspirator, cautiously at first to keep the distilling pot from boiling over. Then full vacuum is applied, and heating begun again. A good aspirator will distill phenylacetone at about 120-130° C. If the water isn't very cold where you live, the temperature will be correspondingly higher. The yield should be in the neighborhood of 100 ml, and the product should be clear to maybe slightly pale yellow. It should have that characteristic strange, chemical, fruity smell of phenylacetone.

For those familiar with phenylacetone, just recognizing its smell, and noting its boiling point is sufficient to confirm its identity. For those not so blessed by experience, one can positively ID the product as a methyl ketone by seeing if it forms a solid bisulfite addition compound. Take about 5 ml of the distillate, put it in a test tube along with at least an equal volume of water saturated with sodium bisulfite or metabisulfite. Shake for a couple of hours. If the product is phenylacetone, a solid, crystalline product will form.

For the clandestine cooker, the recipe as given presents one big problem, and a couple minor ones. The big problem is the large amount of solvent it uses, and the fact that this solvent is anhydrous ether. Anhydrous ether purchased in large amounts is an item of suspicion, and its characteristic smell carries well downwind. Performing this procedure in a populated area would be ill advised.

A possible way around this problem would be to substitute the very common and easily available industrial solvent hexane for the ether. It only costs a couple of dollars per gallon when purchased in drums, and its smell is nowhere near so pronounced as ether. The recycling of this solvent is pretty simple as well, in contrast to ether, which tends to form explosive peroxides. To recycle hexane, one needs only wash it with some water, then redistill it. Hexane forms an

azeotrope with water that distills at 61° C versus 69° C for the pure hexane, so any entrained water will be removed in the distillation leaving dry hexane to distill in the main fraction. Naphtha may also work.

A much less troublesome problem is the need for absolute alcohol in this procedure. One can obtain this as is from chemical suppliers, or if one desires to be very low profile, absolute ethyl alcohol can be made pretty easily from 190 proof vodka.

For example, one can mix equal quantities of 95% ethyl alcohol and benzene, then distill out the benzene as its water azeotrope which distills at 69° C. The residual water in the alcohol can be removed by adding a little sodium metal, then distilling the pure alcohol. As a general rule, one is better advised to try to get pure absolute alcohol. Another potential problem may arise in obtaining potassium metal. It's not on any lists of guilt-implying chemicals, but one might feel uncomfortable ordering it. If so, it can be made pretty easily by use of an electric cell. The same procedure used in *Secrets of Methamphetamine Manufacture* to make sodium metal from sodium hydroxide can be used to make potassium metal from potassium hydroxide. A similar procedure can be found in US Patent 2,480,655. The metal is very nasty reactive stuff, so buying it ready made is greatly preferable.

Chapter Seven
Phenylacetic Acid From Ethylbenzene

The most convenient precursor to phenylacetic acid among unlisted industrial chemicals is its ester, ethyl phenylacetate. A simple hydrolysis of the ester yields this very desirable but tightly controlled precursor to phenylacetone. Consequently, if one wishes to pursue this synthetic pathway, tapping into the perfumers' supply line is the most logical course of action. However, in the future the esters of phenylacetic acid may also come under regulation. Should this come to pass, a simple and high yielding procedure using common industrial materials is a valuable backup. The chromate oxidation of the industrial solvent ethylbenzene to phenylacetic acid fills this role nicely.

The logistics of supply for this alternative route to phenylacetic acid are pretty simple and easy. To set up a factory of meth production yielding around 200 pounds of pure meth prior to cutting, one just needs a 55 gallon drum of ethylbenzene and several hundred pounds of sodium dichromate or several hundred gallons of used chrome plating bath picked up through a waste exchange. For example, a drum of ethylbenzene weighing about 400 pounds will yield a similar weight of phenylacetic acid, up to about 450 pounds or so if all goes well and the chemical god

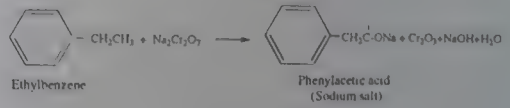
smiles upon your efforts. There are quite a variety of methods for converting phenylacetic acid to phenylacetone given in my book *Secrets of Methamphetamine Manufacture*, and I have personal experience with several of them. For a large scale operation such as one starting with a drum of ethylbenzene, the best method is the tube furnace utilizing thorium oxide catalyst. The procedure is found in the meth book, or *Organic Syntheses*, Collective Volume 1,2, or 3. Look for methyl benzyl ketone in the table of contents. The aerogel modification of the catalyst bed would be most appropriate for such a large scale production, as it allows a greater rate of feed. The glacial acetic acid used to make phenylacetone with the tube furnace is very easily available in drums, and a yield of around 50% is very realizable, yielding a little over 200 pounds of phenylacetone from a drum of ethylbenzene.

To convert the phenylacetone to meth, one is going to need about 100 pounds of methylamine. Methylamine is a hot List I commodity, but is very easily made on a large scale by the method given in my meth book, or by the alternate methods given in this work. Reducing the methylamine and phenylacetone mixture to give meth is best done on this scale using platinum catalyst and the poor man's hydrogenation device described in this book. The yield is nearly quantitative if the catalyst is kept in good working order.

In this manner, a factory could be set up in some industrial site with four work stations — one to convert ethylbenzene to phenylacetic acid, another to make phenylacetone from the phenylacetic acid, another to produce the methylamine, and a final site that turns out meth. Two hundred pounds of meth, cut in half to give 400 pounds, and sold at $5000 a pound works out to 2 million dollars for an investment of a few thousand.

This reaction is pretty simple and cheap to do, but requires a little bit of preliminary engineering and construction. The engineering and construction consists of getting some sturdy steel pipe, welding two or three agitator vanes on the inside of the pipe, and pulling the agitation gearing out of an old washing machine and reinstalling it onto the pipe. It's no great challenge.

This reaction was originally reported in *Journal of Organic Chemistry*, Volume 27, pp. 27-28 (1962). It was found that when ethylbenzene was heated with sodium dichromate in water solution at a temperature of 275° C inside an autoclave, a 90% yield of phenylacetic acid resulted.



Ethylbenzene is churned out in huge quantities by the chemical industry, as it finds use as a feedstock for making styrene and also as a solvent for plastic resins. Any attempt to try to keep track of its commercial flow would be futile. Drums of it are easily available once you give yourself a corporate name.

Similarly, sodium dichromate is heavily used by industry. Its largest and most common use is probably to passivate stainless steel. This process essentially consists of immersing a piece of stainless steel into a vat containing a mixture of nitric acid and sodium dichromate.

The passivated stainless steel is more corrosion resistant than untreated steel. Calling yourself some kind of metal

finishing or polishing operation and getting a shed or something to work in will make ordering a thousand pounds of sodium dichromate pretty easy.

An alternative source of dichromate is a used chrome plating bath picked up on one of the waste exchanges. See *Practical LSD Manufacture* or *Secrets of Methamphetamine Manufacture* for information on waste exchanges. The typical used chrome plating bath will contain 32 ounces per gallon of chromic acid. The exact concentration will come with the analytical sheet. This is a 2 molar chromic acid solution. At this concentration, the great majority of the chromic acid is in the form of the dichromate. To get a sodium dichromate solution, add a 20% solution of sodium hydroxide to the chrome plating bath, slowly and with stirring, until the pH reaches 8 or 9. Then add water until the volume of the solution is double that of the starting chrome plating bath. This results in a .5 molar solution of sodium dichromate. The tramp metals in the bath, such as iron, copper or nickel, will settle out as a hydroxide sludge. Decant off this sludge, and filter to get a clean .5 molar dichromate solution.

The following example is given for a .25 molar size batch, but the procedure could easily be scaled up to at least one molar and beyond by giving allowance for the longer period of time such a larger batch would require to heat up to the proper reaction temperature.

First one starts with a section of heavy pipe, of a volume of about 1500 ml. It should have fine threads on each end. Obtain heavy pipe caps with matching threads for both ends. Most pipe will come with a heavy coating of zinc galvanizing on it. This must be removed by immersing the pipe and caps in 5% hydrochloric acid solution until the rapid fizzing caused by the dissolving of the zinc slows to a

crawl. Then thoroughly rinse the pipe and caps in clean water, and dry them to prevent rust.

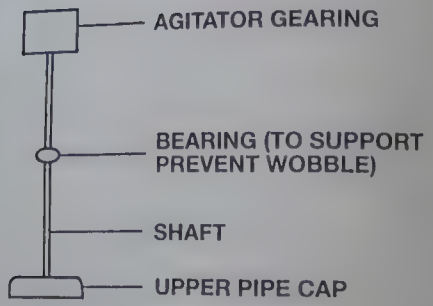
Then agitator vanes should be welded onto the interior of the pipe. There should be three or more vanes, depending upon the diameter of the pipe. For example, a one gallon size pipe section for a one mole batch scale should have four vanes to get good agitation. They should run the entire length of the pipe section, and extend about halfway in from the wall of the pipe to its center point, as shown in the example below (which, of course, features only three vanes):

TOP VIEW

The agitator vanes, of course, should be made of steel, and be clean and free of paint.

Once the vanes are welded in, the bottom cap can be screwed on the pipe, tightened down with a pipe wrench, and preferably welded into place.

Now the cap assembly is constructed. It consists of a shaft welded onto the top of the upper pipe cap. The shaft runs to the washing machine agitator gearing, preferably with a bearing about halfway up the shaft to prevent wobbling as the agitation is going. See the figure on page 60.



To do the reaction, put 1000 ml of water into the pipe, along with 74 grams of sodium dichromate. Alternatively, put 600 ml of water in the pipe along with 400 ml of the .5 molar dichromate solution made from the used chrome plating bath. Next add 30 ml of ethylbenzene. Then screw the pipe into the upper pipe cap, and tighten it with a pipe wrench.

The reaction mixture must be rapidly heated to 275° C. The best way I can think of to do this is to use solder as a high temperature heating bath. Spools of solder can be easily picked up at the hardware store and tossed into a heavy iron pot for melting. Solder melts at about 220° C, and so makes a suitable heating bath for this reaction. The solder bath should be heated to about 300° C, then the pipe reaction vessel should be immersed in it to a depth about equal to the depth of the liquid inside the pipe, and agitation should then

be commenced on the pipe reaction vessel. The solder bath should be of a volume at least equal to the reaction pipe so that it holds a fair sized heat reservoir. The temperature of the solder bath will quickly fall when the pipe is immersed, but it shouldn't be allowed to fall below 275° C.

It will take about 15-20 minutes for a batch of this size to warm up to the reaction temperature of 275° C. A larger batch will take longer to warm up. The geometry of the pipe will also effect the length of time required for the reaction mixture to warm up. A longer, narrower length of pipe presents more surface area, and so warms up faster than a shorter, fatter one.

Once the reaction temperature is reached, continue heating at 275° C with good effective agitation for one hour. The back and forth agitation action must be vigorous enough to force the ethylbenzene, which floats on top of the water and chromate solution, into contact with the chromate. Once it reacts and forms the sodium salt of phenylacetic acid, it freely dissolves in the water. If at the end of this process one should find an organic layer still floating on top of the water, then the agitation was too slow, or not enough time was allowed for the mixture to reach reaction temperature prior to counting off the one hour reaction time.

During the course of the reaction, there will be a steam pressure inside the pipe amounting to hundreds of pounds per square inch. For this reason, the top must be cranked down good and tight, and the pipe must be of heavy construction. As a precaution, don't hover around the batch while it cooks. Also wear a face shield, leather jacket, apron and gloves. Be watchful of globs of solder spinning off the outside of the pipe.

When the one hour reaction heating period is over, the agitation should be stopped, and the kettle of molten solder lowered from the reaction pipe and wheeled away. Next,

cool down the reaction pipe by immersing it in a tub of water. When it has cooled down, the tub of water may be removed, and the pipe reaction vessel opened with a pipe wrench.

The green colored contents should be poured into a beaker, and then filtered to remove the chrome oxide sludge. Rinse the sludge to get any product off it. If time permits, it might be easier just to let the sludge settle to the bottom of the beaker, decant off the liquid for filtering, resuspend the sludge with a little water, and then filter this remnant.

Now to the filtered reaction mixture, add 6N sulfuric acid (this is concentrated sulfuric acid diluted in half with water) slowly with stirring until the pH of the reaction mixture gets down to two or even one. This causes the sodium salt of phenylacetic acid which was produced in the reaction to convert to phenylacetic acid. Phenylacetic acid will dissolve in hot water, but crystallizes out of cold water.

Using a funnel to prevent spillage, transfer the now acidified reaction mixture to a large separatory funnel. Then extract two times with 100 ml portions of toluene. This solvent can be found at the hardware store in the paint section. They may call it toluol.

The extracts should be transferred to a distilling flask, and the glassware rigged for fractional distillation. The first portion of toluene distilling will azeotropically dry the phenylacetic acid. It will distill with a milky appearance. Then the main body of the toluene will distill with a clear color. When most of it has distilled, remove the residual toluene in the product by applying a vacuum.

When the toluene has been distilled out, stop the vacuum and the heating, and while the phenylacetic acid is still molten in the flask, pour it out into a sturdy glass baking dish, or something similar. There it can set up into a hard, white mass that you can chip at when needed. The yield from

this size run will be 25-30 grams. A one gallon size batch will yield over 100 grams.

Now that the basic process has been explained, some further discussion from one learned in this art is called for. The first point to consider has to do with the distillation just done. If one is using home constructed stainless steel distilling vessels as described in the Fourth Edition of *Secrets of Methamphetamine Manufacture*, one would be well advised to devote one set just to this process, and use it for no other procedures. The bottom of the distilling flask could be rounded, and a stopcock with valve welded into the lowest point of the bottom. Then the molten product could just be drained into the baking dish at the end of the distillation. This will keep the mess making with the very smelly phenylacetic acid to a minimum.

The second point has to do with just that, the reeky smell of phenylacetic acid. It smells like you have a whole troop of screaming tom cats pissing away at the walls. The sodium salt of phenylacetic acid doesn't stink, but the free acid sure does. The narco swine are well aware of what that cat piss smell means, so lay down newspapers where some may spill or drip. Then burn the papers! Once on a surface phenylacetic acid will continue to stink forever, almost, and is very difficult to wash away. One washing procedure that does work is to dissolve some lye in rubbing alcohol, and using gloves, of course, sponge the surface that is contaminated. This will produce the non-stinky sodium salt, which also dissolves well in water and so can be scrubbed away.

On the topic of water, if this process is scaled up to where hundreds of pounds of sodium dichromate are being used, some attention to pollution should be paid. The filtered out chrome sludge should not just be dumped into a sewer. Most all places served by sewers in the civilized world have monitoring of the incoming sewer water. These monitoring

stations are located on the sewer mains, and if they pick up loads of chrome coming at them, they are going to look for the source. The trivalent chrome produced by this reaction isn't really bad like hexavalent chrome is, but you can still get yourself unwanted attention by dumping chrome into the sewers. A few pounds nobody will notice. A few hundred is another matter. For a large scale operation, my advice is to drum up the sludge. When the cooking is completed, spend the thousand or so dollars to have it carted away by a hazardous waste firm.

Chapter Eight
Another Low Profile Phenylacetone Factory

In the previous section, a low chemical feed profile phenylacetone factory using ethylbenzene to produce phenylacetic acid was detailed. That procedure is quite practical and workable, but not without its problems. Prominent among the problems attendant to that method are the persistent stink of phenylacetic acid, and the dangers inherent to the use of a high temperature pressure vessel to make the phenylacetic acid.

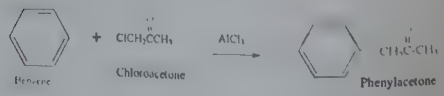
In this section, an equally low profile chemical feed method for producing large amounts of phenylacetone will be covered. It doesn't suffer from the two major difficulties cited for the ethylbenzene process, but it has three areas of concern unique to it. It uses chlorine gas in the process, requiring that the factory be set up in a lightly populated area such as a countryside garage with very good ventilation.

The procedure itself is very nasty and unpleasant to be around. My experience with this process dates to 1979 or 1980, and I can personally attest to the hell which will be experienced without adequate ventilation, and care in handling the materials. Cloroacetone, which is first made from acetone and chlorine, then fed into a Friedel-Krafts reaction with benzene to make phenylacetone, is a most

potent tear gas agent. Even at room temperature, the fumes coming off this material from an opened bottle are enough to clear an unventilated room quickly.

Another problem with this procedure is the seemingly unavoidable low yield of product one gets. The literature states a yield in the low 30s of phenylacetone based upon the amount of chloroacetone used. My experience is that this is probably a little bit on the high side, and that 25-30% is more realistic. The original authors of the procedure tried using a greater dilution with benzene to raise the yield, and had no success. A correspondent wrote once claiming that he had found a way to vary the procedure to get a 50% yield, but he gave no details. This correspondent is known for making inflated claims, besides being a snitch who would sell his mother down the river to save his hide, so judge this claim with caution.

See *Journal of the American Chemical Society,* Volume 62, page 1622 (1940), for the original disclosure of this procedure. Chloroacetone reacts with benzene using anhydrous aluminum chloride catalyst to produce phenylacetone, along with a bunch of tarry by-products.



Into a 5000 ml three necked flask equipped with a reflux condenser protected with drying tube in one neck, a mechanical stirrer in another, and a dropping funnel in the third neck, put 410 grams of anhydrous aluminum chloride, and 1000 ml of benzene. Benzene is a fairly common industrial solvent whose use isn't as widespread as it used to

be before the discovery of its purported carcinogenic properties. Five gallon pails and 55 gallon drums are fairly easily obtained at dealers of industrial chemicals. Look in the yellow pages for dealers, and read over the "War Stories From The Pen" chapter for ordering etiquette. Anhydrous aluminum chloride isn't so commonly used as the hydrated form, but it too should be pretty easy to get at industrial chemical outlets. Avoid dealing with scientific supply houses, and other hotbeds of chemical sales reporting. If they sell List I chemicals, stay away. 5000 ml three necked flasks don't exactly grow on trees these days, but a stainless steel replica coated on the inside of both the flask and condenser with Teflon will give good service. See *Vestbusters* for Teflon and Teflon-based paint ordering and application procedures.

Then with stirring, heat the benzene to a mild reflux boil. Using a coated steel pot, this would be difficult to see. In that case heat the pot in an oil bath maintained at 90° C, and when the neck of the pot leading to the reflux condenser gets hot, the pot is boiling. When the mixture is hot, dropwise add 140 ml of chloroacetone through the dropping or addition funnel. This addition should take between ½ hour to a full hour. When the addition is complete, continue boiling the mixture for an additional 5 hours. The mixture turns an ugly, very dark black shade, and HCl escapes through the top of the reflux condenser while the mixture cooks.

When the 5 hour boil is complete, cool down the mixture. Then slowly add water to the mixture with stirring. A lot of HCl is given off when the water is added, especially at first. Add about one liter of water. By the end of this water addition, evolution of HCl should have ceased, although the mixture will continue to smell like hydrochloric acid. Next add a mixture of 200 ml of water and 200 ml of concentrated hydrochloric acid (30-35% strength) with continued stirring.

This assures that the aluminum salts end up in the water layer.

Next, pour the mixture into a large separatory funnel, and separate off the black benzene layer. The product is in there somewhere. Then take the water layer and extract it four times with 250 ml portions of benzene. Add these extracts to the black benzene layer. Finally, filter the benzene-product mixture.

Now return the filtered benzene product mixture to a 5000 ml distilling flask. Rig this flask for fractional distillation. The fractionating column doesn't have to be anything elaborate, as just a little bit of fractionating power will do. Heat the mixture and distill off the benzene. The first portion will be the benzene-water azeotrope boiling at about 70° C. It will have a milky appearance, and can be reused as the extractant in the previous paragraph. When all the water has been removed the distillate will be clear colored, and the benzene will boil at about 80° C. This benzene can be reused in the production reaction.

When almost all of the benzene has been distilled off (the rate at which it distills over falls greatly), transfer the residue in the distilling flask to a 1000 ml distilling flask or, if possible, a 500 ml distilling flask. Rig this flask for fractional vacuum distillation, and apply a vacuum. The residual benzene quickly comes off. Then apply heat, and vacuum distill the residue. About 90 ml of liquid distills below 123° C at a vacuum of 20 torr. The distillation temperature will of course vary with vacuum strength, so the general rule is to collect the first 90-100 ml of product that distills once the benzene is gone. This is the crude product.

The residue left in the distilling flask will quickly set up into a mass that's next to impossible to remove if it isn't poured out immediately at the end of the distillation. Do so at the earliest possible opportunity once the crude product is

secured. The thin film left clinging to the distilling flask can be removed with solvent, but if the whole mass of tar is allowed to set up, it's next to impossible to remove with solvents.

The crude product should be purified by means of the bisulfite addition product. Phenylacetone will react with sodium bisulfite to form a solid derivative. To make the derivative, take the roughly 100 ml of crude phenylacetone, and put it in any convenient container. Next add 300 ml of a saturated solution of sodium bisulfite or sodium metabisulfite in water. Roughly 30 grams of bisulfite will dissolve in 100 ml of water. Shake or stir violently for a few hours. A thick mass of crystals should form in the mixture. Filter these out, and rinse them with a little solvent such as benzene or toluene. These crystals are the bisulfite addition product of phenylacetone.

To regenerate phenylacetone from this addition product, put them in a flask along with about 800 ml of a saturated solution of sodium bicarbonate in water. About 80 grams of bicarb will dissolve in 800 ml of water. Heat the mixture to boil under a reflux for a few hours. This should completely decompose the addition product to give back phenylacetone. It can be recovered by adding about 100 ml of toluene or benzene to the flask, shaking to extract phenylacetone, then separating the product layer that floats on top of the water with a sep funnel. After the benzene or toluene is boiled away, the residue is phenylacetone. The yield should be around 70 ml.

If there is any evidence that the bisulfite addition product is less than completely decomposed, the phenylacetone should be recovered from the bicarbonate solution by means of steam distillation. The exact technique of doing a steam distillation is covered in any Organic Lab text, and will always be done as part of Organic Lab coursework, so the

details won't be covered here. Just a few tips. A pressure cooker is a good source of steam. Just run a line from the steam vent at the top into the flask. It will take a few liters of water to steam distill about 70 ml of phenylacetone. The phenylacetone is then recovered from the steam distillate by extracting with some toluene or benzene, and the solvent boiled away to give a residue of phenylacetone. It should be transparent with a clear to light yellow color, and have that characteristic chemical kind of fruity smell of phenylacetone. There should be no trace of the chloroacetone.

Preparation of Chloroacetone

While chloroacetone isn't on any federal reporting lists as of mid-1997, it has no industrial usefulness that I know of. That means that the only likely source of it ready made is from scientific supply houses. Let your Uncle be your guide here, and stay away from these sources. One is much better served cooking your own chloroacetone from acetone and chlorine than putting your name on their snitch list. The people who run these supply houses aren't so stupid that they have no idea of the uses to which chloroacetone can be put. Any bottle obtained from them would have to be redistilled before use anyway because the stuff gets black and tarry in storage. If you are going to have to go through that, you might as well make the stuff.

The materials from which chloroacetone is made are very easily available, cheap, and in large quantities. A meth lab project which would make the operator rich beyond the dreams of avarice is easily realized by this production route. The same can be said of the route using ethylbenzene. The choice is one based upon taste, one's choice of persistent stink or a tear gas assault.

Acetone can be picked up at any hardware store, in the paint department. It comes in one gallon pails for under $10 each. If production is going to be scaled up to the point where a lot of these pails are going to be consumed, spread out the purchases to a bunch of hardware stores. I recall seeing a "Most Wanted" type show where a hardware store owner called the cops because the same person kept coming back for all these pails of acetone. Hardware store acetone will contain a percent or two of water which must be removed prior to use, but this is easy to do. The hardware store is still the place to get acetone.

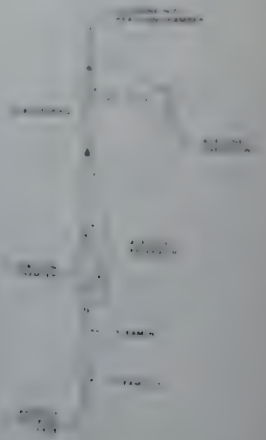
Chlorine is an industrial gas with a variety of legitimate uses. The chemical truck which pulls up to the plant where I work is often carrying cylinders of chlorine. They go to places which use them for water purification, making pool chemicals or to destroy cyanide in waste water from electroplating operations. Fashion your company name accordingly when ordering this material.

Similarly, the sodium bisulfite or metabisulfite used to purify the phenylacetone is an easily available commodity. In smaller packages of a pound or so, one need go no farther than the local brew supply shop. Home brewers use the chemical to disinfect their brewing vessels. Vintners add it to their wine to prevent spoilage. The plant where I work consumes tons of the stuff in the waste water treatment process. We use it to reduce hexavalent chrome to trivalent, which can then be sludged out of the water by adding hydroxide. For this purpose, it comes in 50 pound bags. Obtaining it from an industrial supplier of chemicals is no problem. Once again, see the "War Stories From The Pen" chapter for ordering and pick up etiquette.

The process for making chloroacetone is simple enough, giving a high purity product so long as the acetone and chlorine are free of water. Some simple engineering is

required to assemble the reaction apparatus. The best place to put this apparatus is in a countryside garage, in the central section of said garage. One should work upwind from the apparatus, and have a fan to assist the natural direction of air flow.

See US Patents 2,235,562 and 2,243,484 for more information on this process and alternative construction designs for the reaction apparatus. The assembly shown below is the simplest design I could think of using standard parts.

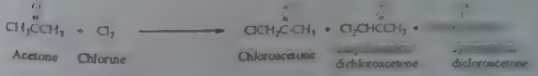


The reaction apparatus is constructed with standard chemical glassware, preferably 24/40 size. At the bottom is a flask where the chloroacetone produced collects. Fitted above it is a condenser, which is heated by passing a flow of

steam through the water jacket. As was mentioned earlier, a pressure cooker is a good source of steam. It's best to put a few pieces of broken glass inside this condenser so that drops of acetone can't just fall into the product flask. Above the heated condenser, one puts a claisen adapter. On one branch of the adapter, put a dropping funnel or addition funnel. Acetone is put in this funnel, and dripped into the reaction apparatus while doing the reaction.

On the other branch of the adapter, put a kugelrohr bulb. This is the reaction chamber. Above that is placed two condensers. Cold water is run through them to condense acetone vapors. Above that, a stillhead is put into place. Through the top of the stillhead run a narrow piece of glass tubing down to the center of the reaction chamber. The glass tubing carries chlorine gas into the reaction chamber. It should be of narrow diameter so that the condenser will not easily flood out with acetone unable to flow downward to the reaction chamber. Finally, a standard distillation receiver assembly is attached to the stillhead to catch any acetone vapors which make it out the top of the condensers, and also to provide an outlet for the HCl gas produced in this reaction.

Chlorine reacts with acetone vapors to produce three possible products:



A molecule of HCl is also produced for each chlorine atom that adds to the acetone.

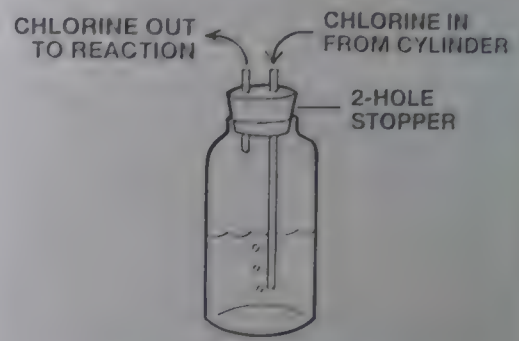
It is pretty easy to assure that the vast majority of the product is the desired chloracetone. The byproduct sym-

[illegible] dichloroacetone is produced when too much chlorine is introduced into the reaction chamber. One can keep the reactants in balance simply by watching the color of the vapors in the reaction chamber. A faint shade of green is desirable. If the color gets darker green, the rate of input of chlorine gas should be decreased, or the rate of input of acetone increased.

The byproduct unsymmetrical dichloroacetone is produced when water is present in either the acetone or chlorine. This byproduct is very troublesome because it boils at the same temperature as chloroacetone. As a result, removing it from the product by distilling is impossible. One must therefore make sure that both the acetone and chlorine are free from traces of moisture.

The most common method of removing the traces of water from the acetone is to use Epsom salts as a drying agent. As they come from the store, the Epsom salts ($MgSO_4\ 7H_2O$) contain a lot of absorbed moisture. To use them as a drying agent, this water must be baked off. Get a glass baking dish, and put a layer of Epsom salts about ¼ inch thick into the dish. Bake it in an electric oven at 400-450 F for an hour or [illegible] the water. Then turn off the heat to the oven [illegible] down to where you can just handle the dish with bare hands, and pour the baked Epsom salts into a glass jar, and screw the lid back on the jar. This will keep the baked Epsom salts from reabsorbing water from the air. To dry [illegible], put about 100 grams of these baked [illegible] a gallon pail of acetone. Close up the cover [illegible] pail, and periodically shake it around, or tip it [illegible] sides. Let the Epsom salts work for about a [illegible] let them settle. The acetone should be directly [illegible] the pail to the acetone reservoir with help from a funnel when the reaction is done. It's best to have filter paper in the funnel to catch any suspended Epsom salts.

The chlorine gas feed for the reaction should similarly be dried. This is done by passing the gas into a wash bottle containing some concentrated sulfuric acid as shown below. This not only assures that the chlorine is free of moisture, it also is an easy visual way to gauge and adjust the rate of flow of chlorine into the reaction.



Glass tubing is used in the wash bottle. Plastic tubing can be used as the gas line from the cylinder and out to the reaction.

To make chloroacetone, first begin passing steam through the lower condenser, marked steam in and steam out in the diagram. Then start the flow of cold water through the other three condensers. Next add a little bit of acetone, like about 5-10 ml, into the apparatus through the acetone reservoir. It will vaporize as it flows down the claisen adapter into the heated condenser. The vapors will condense in the upper cold condensers, flow back down, and cycle like that.

Next, begin a new flow of ammonia from the diffuser to the flask and add enough of more to the mixture to fill up the flask once and then make no way per the reaction container. After a minute or slight pressure can will be released. Disconnect all from, and the liquid will flow down the vent tubes underneath the flask that collects the product. Disconnect this at this C and re-add ammonia as was done on the previous flask.

Once the ammonia stock is empty in the reaction apparatus, a new flow of ammonia from the reaction source begins. The rate of addition of ammonia should be such that the acid water solution does not overflow the flask. The rate of addition of ammonia should be adjusted so that a faint greenish color is maintained in the mixture and surrounding reaction area of the solution. A darker green color indicates that too much ammonia is being added relative to the amount of nickel salt.

The water layer color serves the purpose of determining when enough ammonia has been added. The rate of addition of ammonia and ammonia are measured to maximize high-purity nickel production.

When enough ammonia has been added, turn down the flow of ammonia. Disconnect the tubing at the diffuser and remove the flask of ammonia. Discharge production from each flask, or as stated in the previous procedure will always be a greater height.

The product should be treated as a very toxic and should only be handled by individuals of known to know. The handling should be done with the fuming hood and the exact safety measures followed. Because of the product forming at or the top based, ammonia mixture of product is that has been removed and the upper. Public

gloves are required as well. The water layer from the flask solution, which can be thrown away. The product layer should be poured into a beaker that is 2 times by bubbling of calcium chloride with nitrogen. Calcium chloride can be found at the store as 50 pound bags. It is used to melt ice. Once the bag is opened it will soak up water from the air and melt. Calcium chloride used for this purpose should form the beaker at the start to about 400 F gas like Epsom salts, when mixed in a sealed container will give heavily use the calcium chloride with nitrogen and a water layer separate from the product.

The drying hood separate off the water layer from the product is ready for distillation. A thermometer should be used like the glass pyrex calcium sensor to the Fourth Edition of Barron or Mallinckrodt Manufacturer. Place thermometer with a rubber C as normal pressure. Some vacuum is preferred for distilling the methadone, with all the vacuum delivered from a vacuum aspirator. The strong vacuum will lower the boiling point so much and make the methadone difficult to control. A vacuum of 30 torr will result in a boiling point of about 90 C. No stronger vacuum should be used, as the methadone. The pressure should be kept at a consistent glass container and heated to the point until. The long chemical of storage will result in the product boiling point.

Chapter Nine
A High Yield
Phenylacetone Recipe

At the time of this writing, the acid chloride of phenylacetic acid, phenylacetyl chloride, is not included in the list of reportable chemical transactions. No doubt, this oversight will soon be corrected so that the world can be saved. Nevertheless, the acid chloride of phenylacetic acid is a very good precursor for the manufacture of phenylacetone. If one is in a position where the other chemicals required to do this reaction are easily available, then this is one of the best procedures for making phenylacetone.

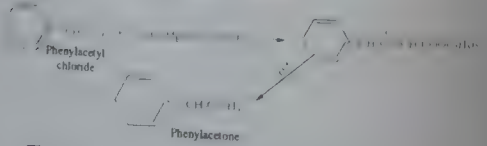
Let's start with the making of the acid chloride of phenylacetic acid, phenylacetyl chloride. Directions are found in the *Journal of the American Chemical Society*, Volume 70, pp. 4241-45 (1948). In a 500 ml round bottom flask, place 136 grams of phenylacetic acid, and 110 ml of thionyl chloride. The last time I checked, thionyl chloride was on the California list of controlled chemicals. Other states may also require reporting of its sales, so check first. It's far better if this material is just laying around on some shelf for the taking.

Now attach a reflux condenser to the flask, and boil at reflux. HCl and SO_2 will be given off, so a plastic tube attached to the top of the condenser leading to fume disposal is required at first. Then as the reaction nears completion, the

fume production slows and then stops. One can determine when it has stopped either by sniffing at the top of the reflux condenser, or preferably by moistening some pH paper and holding it at the top of the condenser. When it doesn't give a quick and strong acid reaction, then the process has reached completion.

Next rig for fractional distillation. The unreacted thionyl chloride can be distilled off at a boiling point of 76° C, then a vacuum is applied to get a 95% yield of phenylacetyl chloride which boils at 95° C under a vacuum of 12 torr, 105° C at 24 torr, and correspondingly higher temperatures at less powerful vacuums.

The procedure for making phenylacetone in about 70% yield from phenylacetyl chloride can be found in the *Journal of the American Chemical Society*, Volume 68, p. 1386 (1946). The following reaction is done:



The procedure for turning phenylacetyl chloride into phenylacetone is fairly simple, but it requires that one use magnesium turnings, which are on the California list of restricted chemicals. Also, anhydrous ether is needed as the solvent. The smell of ether carries well downwind, and ether poses a fire hazard as sparks can easily set the fumes aflame. Additionally, anhydrous ether isn't a particularly low profile substance to obtain. This method is only recommended to those who have easy access to the chemicals. The low profile phenylacetone prep from phenylacetic acid is the use of a tube furnace packed with thorium oxide catalyst.

To do the reaction, a 2000 ml round bottom flask which has been baked to remove traces of water is loaded with 26.75 grams of similarly baked magnesium turnings. Then add 25 ml of absolute alcohol and 2.5 ml of carbon tetrachloride. Start magnetic stirring, and the reaction starts almost immediately. Allow the reaction to go a few minutes, then add 375 ml of anhydrous ether slowly. Heat the mixture with steam, and add a solution of 176 grams of diethyl malonate in 100 ml absolute alcohol and 125 ml anhydrous ether at such a rate that a rapid reflux is maintained. Apply steam heat if needed to keep the reflux going during the addition. Then reflux for 3 hours or until the magnesium dissolves.

To the clear solution that results when the magnesium has dissolved, add with vigorous stirring 132 ml of phenylacetyl chloride dissolved in an equal volume of anhydrous ether. Reflux for ½ hour, then cool the solution.

The cooled solution is next poured into a large sep funnel, and 500 ml of a 10% by volume solution of sulfuric acid in water is added. The mixture is shaken for a few minutes, then allowed to stand. The water layer in the bottom of the sep funnel is next drained out of the sep funnel. Check the water to make sure it is still acid. If not, shake the ether layer in the sep funnel with an additional 150 ml of sulfuric acid solution, drain it off and check it for acidity.

Now pour the ether solution into a large filtering flask, or other container to which a vacuum can be applied. Extract the sulfuric acid solution with a couple hundred ml of ether, and add this ether extract to the vacuum evaporation container. Apply a vacuum to evaporate off the ether.

To the residue left in the evaporation container when the ether is gone, add a mixture of 300 ml of glacial acetic acid, 38 ml of sulfuric acid and 200 ml of water. Reflux for five hours. Phenylacetone has now been formed.

Chill the mixture in an ice bath, and add a solution of a pound of lye dissolved in five pints of water. Shake the mixture strongly for about five minutes, then let it cool down some more in the ice bath.

Next add about 300 ml of toluene, and shake the mixture for a couple minutes. Separate off the toluene layer floating on the top. It contains the phenylacetone. Pour it into a distilling flask. Rig for fractional distillation, and distill first at atmospheric pressure. The toluene-water azeotrope will distill first at 85° C, followed by pure toluene at 110° C. When almost all of the toluene has distilled out, apply a vacuum to remove the rest of the toluene, and then distill the phenylacetone. A good aspirator will distill phenylacetone at about 110° C to 120° C. The exact boiling point will depend upon how strong the applied vacuum is. The yield is about 100 ml of phenylacetone.

A few years after the previously mentioned paper was printed, a guy named Bowman reported on an improved recipe. See *Journal of the Chemistry Society of London*, pp. 322-25 (1950). In this improved procedure, he replaced the anhydrous ether with benzene. Just this is a big improvement. He further went on to improve the procedure by distilling off the unreacted excess alcohol from the first stage of the procedure as the benzene-alcohol azeotrope which distills at 68° C, prior to going on to adding the acid chloride. This eliminates a side reaction of the acid chloride with the alcohol. He also went on to tinker with the acid hydrolysis step to make the conversion to the ketone more complete. While he didn't use phenacetyl chloride in his experiments, he used many other acid chlorides with uniformly excellent results. By using this improved procedure, the yield can be expected to increase from the low 70% range up into the eighties or nineties. Serious experimenters would be well advised to try his improved

method when making phenylacetone by this route. Try using toluene instead of benzene. All the other problems with this route concerning availability of needed chemicals such as the acid chloride and the magnesium turnings remain, however.

Chapter Ten
Electric Phenylacetone

In *Chemistry Letters*, pp. 1021-24 (1977), a fairly simple method is given for the direct conversion of benzyl chloride to phenylacetone. The standard methods for converting benzyl chloride to phenylacetone are first to react with cyanide to get benzyl cyanide, and then benzyl cyanide is either converted to phenylacetic acid, which is then used to make phenylacetone, or the benzyl cyanide is condensed with sodium metal and ethyl acetate in anhydrous alcohol to give an intermediate which yields phenylacetone when heated with sulfuric acid.

The method revealed in the *Chemistry Letters* article is a direct conversion of benzyl chloride to phenylacetone, in a yield similar to the standard procedures outlined above. This method uses acetyl chloride as the chemical which links up with benzyl chloride to form phenylacetone. This chemical is under no types of regulation or control as of this writing, in contrast to benzyl chloride which is on a less stringent federal reporting list. This reporting list requires that chemical transactions involving more than 1 kilo of benzyl chloride be kept on record by the vendor for a couple of years, and supplied to the feds on request. Benzyl chloride is

a very good material to bring across the border from Mexico, as imports of less than 4 kilos need not be reported.

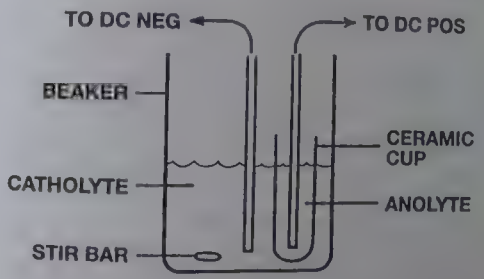
This method uses a lot of acetonitrile as solvent, and uses tetraethylammonium p-toluenesulfonate as the electrolyte. The acetonitrile can no doubt be easily recycled back into the process, but the large dilution holds down the amount of product which can be obtained from a given size reaction apparatus. The electrolyte isn't exactly your common garden variety chemical that any experimenter would want to have. Further, both of the reactants, benzyl chloride and acetyl chloride are foul materials that are best suited to be tear gasses, and not things that you would want to play around with in some unventilated basement. Weigh all these factors in judging the desirability of this process.

Graphite electrodes are needed to do this reaction. Rod shaped graphite electrodes can be obtained from welding supply shops in diameters up to about ½ inch. Better still would be flat sheets of graphite, as they present more surface for a given length, and it's also easier to keep the current density constant over the active surface of the electrode. More surface means more current can be passed, allowing for quicker reactions. Keeping the current density within a prescribed range of values makes for a cleaner product. Flat sheets of graphite can be obtained from plating supply companies.

To do a .2 mole sized batch, which will yield about 9 ml of phenylacetone, one starts with a 2000 ml beaker and a porcelain or other ceramic cup. Remove all the glazing off both the inner and outer surface of the cup with sandpaper or sandblasting. Without removing the glaze, the cup can't pass current. The cup serves the purpose of preventing contact of the reaction solution with the anode. For the cell to be able to pass current, the porcelain or other ceramic should be no more than 3 or 4 mm thick. If it is thicker than this, then an

impractical amount of voltage would have to be applied. Also, a pretty fair amount of heat is generated by the resistance of the ceramic. In effect it is a heater for the solution. Too much resistance would translate into more heat than could be carried away by a water bath on the outside of the beaker.

Assemble the reaction cell as shown below:



Basically what is done is the ceramic cup is placed inside the beaker, then a graphite anode is placed inside the cup, and a similar size graphite cathode is positioned outside the cup, close to but not touching its outer wall. Electrical contact to both the anode and cathode must be secure to prevent sparking. Since acetonitrile is quite flammable, sparks due to bad contacts would be disastrous! A convenient way to make contact with graphite is to turn a brass screw into the graphite, and then wrap and clamp down copper wire around the screw. I picture the anode and cathode extending above the top of the beaker, but this isn't necessary. The tops just need be above solution level.

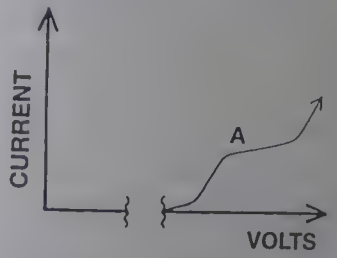
Next put about 640 ml of acetonitrile (anhydrous) in the beaker, and dissolve 15 grams of tetraethylammonium p-toluenesulfonate in the acetonitrile. This is the catholyte. Then add acetonitrile to the ceramic cup until its level is about equal to the catholyte. Record how much acetonitrile was put into the cup, then add tetraethylammonium p-toluenesulfonate to the cup so that its concentration is equal to the catholyte. This is the anolyte. Stir it around to mix it in.

Now to the catholyte in the beaker add 25 ml of benzyl chloride, and 50 ml of acetyl chloride. Begin a rapid stir of the catholyte, and begin to apply voltage to the two electrodes. About 90 to 120 volts DC will be required to break through the ceramic cup, so a DC arc welder is the most convenient power source. The graphite cathode inside the beaker is connected to the negative pole of the welder, and the anode in the cup is connected to the positive pole of the welder. An amp meter must be put in line on the wiring, and it must be sensitive and accurate. One can be had at Radio Shack for about $50.

As the voltage is increased, at first there will be no reading of current flow on the amp meter. Finally, once the insulative effect of the ceramic is overcome, some current will begin to flow. How much more to turn up the voltage output is the next topic which must be addressed. The *Chemistry Letters* article uses a standard calomel electrode to give an absolute measurement of how negatively charged the cathode is. They run the reaction at -2.1 to -2.4 volts. To simplify, let's convert this to current density, which can be read right off the amp meter. It's reasonable to conjecture that the cathode in the cell described in the article had an area of about 2 square cm, given their batch size.

From the time it took them to pass the required amount of current, their current density on the cathode was about .1

amp per square cm. If a graph is made when doing a reaction of current flow through the cell versus applied voltage, one should get something that looks like this:



Once the electricity manages to break through the ceramic cup, the current flow should gradually increase with voltage, until the voltage is increased enough for the reaction to occur. Then a steep rise in current called the reduction wave is seen. After the wave, the current again gradually increases with voltage until the next possible reaction, such as reduction of the solvent, occurs. Then another wave is seen. One wants to set the voltage at the crest of the first wave, labeled A in the graph. This will probably be about 5 volts beyond the point where current first begins to flow. The current density on the cathode will probably be about .1 amp per square cm. In calculating the surface area of the cathode, only measure the side facing the ceramic cup, as the back side is shielded from current flow. As current flows, the following reaction occurs:

Two electrons are needed for the reaction, but four are used in the procedure given in *Chemistry Letters* because of inefficiency in the process. If one's cathode measures 10 square cm, allowing a current of one amp, then a .2 molar batch requires 77,200 amp-seconds, or 21.4 hours at a current of one amp. A 20 square cm cathode carrying 2 amps would require 10.7 hours for a .2 molar batch.

The heat produced must be carried away by keeping the reaction beaker surrounded with cold water. The reaction mixture shouldn't be allowed to rise above room temperature. A big batch might even require that cooling coils be immersed in the reaction mixture in addition to cooling the outside of the beaker.

When the required amount of current has passed, the ceramic cup is removed, and the anolyte is poured into a container to be saved for future use. To get the product, the article recommends pouring the catholyte solution into about an equal volume of saturated salt solution in water. This brine solution was next extracted with three large portions of ether, and after evaporating the ether, the product was distilled.

A more practical isolation procedure would seem to be to distill off the acetonitrile. Using a cheap vacuum pump, such as an aquarium pump, should lower the boiling point of the acetonitrile from about 80° C to around 50° C. With ice water in the condenser, it should be easy to condense all the acetonitrile at that boiling temperature, and thereby save it

for reuse. When most of the acetonitrile has distilled off, then following the procedure of pouring the residue into saturated brine, extracting and distilling the product would seem to be much more efficient. The yield should be about 9 ml of phenylacetone, which is comparable to the amount of phenylacetone which would be obtained from 25 ml of benzyl chloride by using the standard procedures.

Chapter Thirteen
The Poor Man's Hydrogenation Device

In many cases, catalytic hydrogenation is the quickest, cheapest, most low profile and highest yielding method for a clandestine chemist to use to produce a desired substance. For example, to make MDMA or meth from their corresponding phenylacetones, by far the quickest, cleanest, and best method is the reductive amination using platinum catalyst. Further, many of the best and quickest methods for converting ephedrine, pseudoephedrine or phenylpropanolamine center upon using catalytic hydrogenation and a palladium catalyst to yield meth or amphetamine. The details of all these processes, along with plentiful references back to the original scientific literature can be found in the Fourth Edition of *Secrets of Methamphetamine Manufacture.*

In that book, I suggested the use of a champagne bottle as a hydrogenation vessel. This construction design has several advantages. The bottle is built to withstand the pressures of hydrogenation done at up to 40-50 psi, the glass is quite inert to the action of corrosive chemicals, and the glass allows the passage through it of magnetic force, which allows magnetic stirring of the contents. This greatly simplifies the hydrogenation process, as no shaker device has to be constructed.

There are serious drawbacks to this design choice, however. First and foremost among these shortfalls is that processes which involve heating the contents of the hydrogenation vessel can't be done in a champagne bottle, as the glass isn't meant to take heat and will fracture. Under a pressure of 30 pounds or so, this is a dangerous situation. Coating the outside of the bottle with fiberglass resin, as suggested in the meth book, helps to minimize this danger, but the danger is still not eliminated.

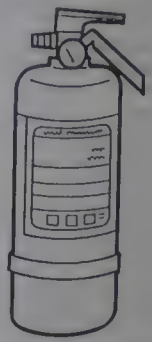
A further deficiency of the champagne bottle design is that it is difficult to attach gas line fittings to the bottle. There just isn't an obvious place on the spout of the bottle where the gas line can be attached and tightened down with a pipe clamp to prevent leakage. Since the bottle is made of glass, there is a limit to how tight such connections can be made.

A couple of years ago, a correspondent who calls himself the Iceworm wrote with his solution, which he calls the "Poor Man's Hydrogenation Device." I find his design to be superior, and worthy of passing on to you. It allows for heating the reaction contents, easy and secure attachment of gas lines, and magnetic stirring. This design comes with my highest recommendations.

The sole shortfall of this design is its vulnerability to caustic chemicals like hydrogen chloride, sulfuric acid and sodium hydroxide. This vulnerability can be overcome by coating the inside of the reaction bottle with Teflon-based paint such as Xylan. See *Vestbusters* for details on how to order and apply this very useful material.

The Poor Man's Hydrogenation Device is a fire extinguisher, the bottle of which is made of aluminum. It is very important that aluminum be used rather than steel, as this allows magnetic stirring. The tech sheet on the extinguisher should state the material of construction, but if one is just shopping, a magnet will reveal what it is made of.

Steel will, of course, attract the magnet, while aluminum will have no action. The 5 pound capacity extinguisher is the best choice. This will easily hold the pint or so reaction size which is about the maximum that can be vigorously stirred magnetically.



To turn this extinguisher into a hydrogenation device, one first discharges its contents. Screw off the top, and clean everything out completely. Next toss a magnetic stirring bar into the bottle, and watch to make sure that it spins properly. Failure to spin well could indicate that the bottle is made of a non-magnetic stainless steel. If caustic chemicals are going to be used in the device, it should then be coated on the inside with Teflon-based paint, and bake cured. A couple of preliminary coats with the low temp bake cure cycle, followed by a final coat with the high temp bake cycle would be the best metal protection. Follow the warnings in *Vestbusters* about excessive coat thickness to avoid bubbling and cracks.

Following coating, the extinguisher is ready to live its new life as a hydrogenation bomb. Pour the mixture to be

hydrogenated into the bottle, along with catalyst and magnetic stir bar. Screw the cap back on and it's ready to go! The discharge nozzle allows for easy attachment to a vacuum source to suck the air out of the device, and similar easy attachment to the hydrogen tank for pressurization. Be mindful that you have to pull on the hand lever to open the valve to both suck the air out, and add hydrogen. The extinguisher even comes with its own pressure valve to monitor internal pressure. This makes keeping track of the progress of the hydrogenation very simple. Sources of leaks are eliminated. A pressure of up to a couple hundred pounds is possible with this device. This is way more than one will need using platinum or palladium catalysts. Periodic checks between batches on the integrity of the Teflon coating inside the bottle are highly recommended when exposing the bottle to caustic chemicals. Ammonia, amines and phenylacetones won't bother aluminum, but more aggressive chemicals will. A dentist's mirror, and small flashlight are desirable tools for these checks.

To heat this hydrogenation vessel, just sit it in an oil bath in a beaker, and heat it on your hot plate-stirrer. It's all very simple with this device. The only area in which it lacks is that the use of Raney nickel is still not so simple. Since this catalyst is magnetic, magnetic stirring is impossible. A way around this is revealed in this book. See the chapter on the Raney nickel cathode (Chapter Three) for a simple, table-top method of using Raney nickel to great effect in making MDA and the other psychedelic amphetamines from their phenylacetones.

Chapter Fourteen
"I Want Some Methylamine!"

To make MDMA, one absolutely has to have methylamine. Similarly, to make meth from phenylacetone, methylamine is a required chemical. As a result, methylamine has been a hot item with the narco swine since well back in the late seventies. Methylamine now sits on the notorious List I, and is a definite "do not purchase or do business with people who sell it" item.

Through the various editions of *Secrets of Methamphetamine Manufacture*, a procedure taken from Organic Syntheses for making your own methylamine has been given. This procedure is quite practical, and gives reasonably good results. Now other processes will be given that offer advantages of their own, along with their own process specific disadvantages. Pick and choose for the method best suited for your situation.

Method One — The Pipe Bomb

This method is taken from US Patent 2,085,785. It offers good yields of methylamine in a shorter reaction time than that required using the procedure from *Organic Syntheses*.

Also, the chemical glassware "kit" isn't needed to make methylamine by this method. A heavy steel pipe reaction vessel and a heater will make the methylamine. Then a good aspirator for vacuum, and a vacuum evaporation chamber are required to recover the crystals of methylamine hydrochloride.

The method has the further advantage of replacing the formaldehyde used in the *Organic Syntheses* process with methanol. Both of these chemicals are easily available from suppliers of industrial chemicals in both 5 gallon pails and 55 gallon drums, not to mention in smaller amounts from hobbyist mail-order chemical outlets. The difference comes in that methanol (wood alcohol) is even more widely used than formaldehyde. You may even be able to find it on the local hardware store shelf, or get them to order you a pail of it. You just can't pass up hardware store chemicals when they are available. HEET is fairly pure methanol.

Both of these methods use ammonium chloride as the other ingredient to make methylamine. This material is easily available from industrial chemical suppliers in 50 pound bags, and in smaller amounts from "hobbyist" chemical outlets. A common use for larger quantities is to slow the melting of snow on ski slopes or sled runs. Craft a company name accordingly.

To produce methylamine hydrochloride by this method, one starts with a section of heavy steel pipe of about 2 quart volume. It must be threaded on both ends, and matching screw-on end caps obtained. Steel pipe is generally heavily galvanized with zinc. This must be removed by immersing the pipe and end caps in 5% HCl solution until the initially vigorous fizzing slows to a crawl. Then rinse the pipe thoroughly in clean water to remove residual acid.

Now screw the cap on the bottom of the pipe, and tighten it down with a pipe wrench. It's best to then weld this cap into place.

Next put 400 ml of methyl alcohol (methanol) into the pipe, along with 535 grams of ammonium chloride, 400 ml of water, and 2.5 grams of ferric chloride. This common chemical often comes as a 40% solution in water for industrial waste water treatment purposes. About 5 ml of this solution would be plenty for the purpose. Mix the ingredients thoroughly, then screw on the top cap. Tighten it down with a pipe wrench as well.

The reaction mixture must be heated at 290° C to 300° C for about two hours. To reach this temperature, the same procedure as given in Chapter Seven should be used. A pot filled with solder should be heated until it melts at about 400° F (205° C). The pipe should then be put in the pot, and the temperature of the pot raised to the desired temperature. Maintain this temperature for a couple of hours. A pressure of hundreds of pounds per square inch will develop inside the pipe, so don't hover around it while it cooks.

When the cooking period is over, turn off the heat and remove the pipe from the pot before the solder solidifies. When it is cool, the cap can be removed, and the contents poured into a large filtering flask. The main reaction product is methylamine hydrochloride, along with some unreacted ammonium chloride and methanol, and some dimethylamine hydrochloride byproduct. The ferric chloride catalyst will color the mixture reddish brown.

To get pure methylamine hydrochloride, the mixture should be evaporated under a vacuum. Just stopper the top of the large filtering flask, and apply aspirator vacuum and heat. As the amount of crystals formed during the evaporation gets copious, they should be filtered out and kept in a glass jar with lid. The mother liquor should then be returned to the

filtering flask for more evaporation. This process should be continued until the volume of the mother liquor reaches between 100-200 ml.

Next recrystallize the collected crude crystals of methylamine hydrochloride. This process will remove ammonium chloride, ferric chloride and dimethylamine hydrochloride from them. Clean out the large filtering flask, then put all the collected crystals in it. Add about a quart of 190 proof vodka to the flask, and heat the contents using a boiling hot water bath. Swirl the contents of the flask around as it gets hot. When it is hot, allow the crystals to settle, then decant off the vodka solution into another flask, and filter the vodka out of the settled crystals. Combine the vodka from the decanting and filtering, and cool it down in the freezer. Return the crystals to the filtering flask.

When the vodka gets cold, crystals of pure methylamine will form. Filter them out, and store them in a glass jar with a lid. Return the vodka to the filtering flask to repeat the heating/decanting/filtering/cooling and crystal collecting process. After a few runs through this process, all of the methylamine hydrochloride will be leached out of the ammonium chloride which remains undissolved in the vodka. The ferric chloride will remain dissolved in the vodka, and should stay completely out of the product crystals. Ditto for the dimethylamine, which shouldn't form in enough quantity to reach saturation in the vodka. The yield should be over a pound of methylamine hydrochloride crystals.

It's a good idea to check for the presence of dimethylamine hydrochloride in the product. This is done by taking a sample of the product and placing it in a beaker. Add enough chloroform that a slush is formed. Stir it around for a few minutes, then filter. Allow the chloroform to evaporate away. If crystals form from the chloroform, this is dimethylamine

hydrochloride. If there is a significant amount, the entire product should be rinsed with chloroform.

If a person has difficulty getting ferric chloride, it can be easily made. Take iron or steel filings, and put them in a beaker. Add 15% HCl, about 20 ml per gram of iron, and heat. The iron will dissolve as ferric chloride. One gram of iron will make 3 grams of ferric chloride. If there is any trace of blue or green color in the resultant solution, pass a stream of air through the solution for about ½ hour to oxidize all the iron to the +3 state. The correct solution color is yellow or brownish yellow. Then boil off most all the acid, until the steam no longer gives an acid reaction with pH paper. Some additional water may have to be added to get all the acid out during the boil down. Don't evaporate the liquid completely away, as overheated ferric chloride will decompose.

Method Two — From Methyl Iodide

This method gives satisfying yields of very pure methylamine hydrochloride, but it requires that one have methyl iodide in addition to hexamethylenetetramine. The latter is easily made from formaldehyde and ammonium hydroxide. See *Home Workshop Explosives* in the RDX chapter for the recipe. An additional problem with this recipe is that about a week of reaction time is needed. That aside, it's very easily done and is recommended if the required materials are at hand.

To make methylamine hydrochloride by this method, dissolve 140 grams of hexamethylenetetramine in 1500 ml of 190 proof vodka. It will be necessary to heat the vodka in a hot water bath to get the hexamethylenetetramine to dissolve. As a result, one should choose a reaction vessel which isn't

going to be infiltrated with the steam. A 2000 ml volumetric flask, or one gallon glass jug are good choices.

When the hexamine has dissolved, add 142 grams (63 ml) of methyl iodide. Stir this substance in well, then stopper the reaction flask and set it aside for about a week. During the course of this week, a crystalline precipitate will form. This crystalline precipitate should be completely finished forming at the end of one week standing.

Now a magnetic stir bar is placed in the reaction vessel and, with stirring, a stream of dry HCl gas is passed into the reaction solution. See *Secrets of Methamphetamine Manufacture*, Chapter Five for details of putting together your own dry HCl generator using salt and sulfuric acid. As the HCl passes into the reaction solution, the crystalline precipitate which had formed will dissolve, and new crystals of ammonium chloride will form. Continue passing dry HCl into the solution until the solution is saturated with HCl. This can be determined by drawing out a sample, and putting it in a beaker. If it wafts off white fume clouds of HCl, and smells very strongly of HCl, then it is saturated.

The mixture is next filtered to remove the ammonium chloride. The vodka filtrate contains about 50 grams of methylamine hydrochloride. It is recovered by boiling off almost all of the vodka under a vacuum. The resulting crystals of methylamine hydrochloride are then mixed with about 2 times their weight of 50% sodium hydroxide solution. (Caution! Mix slowly with ice bath cooling and lots of stirring to avoid losing product.) The resulting free base solution can be used as a source of methylamine by heating the mixture and piping the methylamine gas into a reaction solution. See Chapter Four of *Secrets of Methamphetamine Manufacture* for a typical glassware set up to do this. Alcohol chilled with an ice-salt bath will catch the stream of methylamine produced by this procedure. This alcohol solution of methylamine free base can then be used in reductive hydrogenation methods of producing MDMA or meth.

In other procedures, the methylamine hydrochloride can be used directly as is. For example, when using aluminum foil amalgamated with mercuric chloride as the reducer, one just adds the methylamine hydrochloride to the alcohol solution. Then about twice its weight of 50% sodium hydroxide solution is mixed in to give free base plus some salt and a little excess sodium hydroxide. The presence of both salt and some excess sodium hydroxide in the reaction mixture is helpful to the process.

Chapter Sixteen
More Bromosafrole

In my books *Secrets of Methamphetamine Manufacture* and *Practical LSD Manufacture*, I detail an original recipe of mine for making bromosafrole, a useful precursor to MDA and MDMA, from sassafras oil and 48% HBr. From reading my e-mail, I see that some people are having problems recreating my original research. Sorry that I didn't immediately answer the e-mail. The narco swine now populate cyber space, and just carrying on a conversation with them will land you in the slam as part of their "conspiracy." Funny how conspiracies are ridiculed unless they want to charge you as being part of one. Then conspiracies are everyday matter of fact things. I read a story in *Hustler* about a guy doing 33 years for sharing twisted sexual fantasies with these on-line slugs, freedom of speech in the land of the free, huh?

Problems in doing this process stem from a small detail that I thought was of no consequence. I state in the directions that the sassafras oil and glacial acetic acid and 48% HBr are placed in an "appropriate size flask." Experience has shown that not just any flask will do.

The proper flask for doing the reaction in is an Ehrlenmeyer flask of a size chosen so that it will be nearly full when filled with the reactants. Into the Ehrlenmeyer flask put

one volume of sassafras oil, one volume of glacial acetic acid, and two volumes of 48% HBr. Begin strong stirring, and pass a stream of dry HCl through the solution. The stream of HCl bubbles should be introduced at the bottom of the flask near the stirrer so that they are sucked into the vortex. The inward sloping walls of the Ehrlenmeyer flask act to slow the ascent of the bubbles, and provide a long column height of liquid for the bubbles to pass through. A plug of glass wool stuffed into the neck of the flask extending a few cm down into the solution will get even more play out of the HCl bubbles.

The drying of 48% HBr with dry HCl is a surface phenomenon on the HCl bubbles. Dry HCl will quickly absorb two molecules of water at least from the HBr solution. Some will also go into solution to raise the ionic strength of the solution. The time of contact of the bubbles with the reaction solution has been shown to be very important. If one's magnetic stirrer isn't strong enough to suck in and whip around the HCl bubbles, switch to a mechanical stirrer. Breaking up the bubbles greatly increases their surface area.

The amount of dry HCl produced from dripping about 100 ml of sulfuric acid onto a bed of salt moistened with hydrochloric acid is about enough to do a batch containing 50 ml of sassafras oil. The smell change of a sample, from candy shop sassafras oil to phenylacetone-like, is the indicator of enough HCl. Also take care that the ice bath keeps the reaction cooled to below 10° C.

Method Two

An obvious fan who calls himself Pugsly has posted this method for making bromosafrole on the Internet, and it has been getting rave reviews from people trying it. It's very

simple to do, uses simple chemicals, and gets practically 100% yields. The practical up shot of this recipe is that the so-called pipe bomb method of making MDA and MDMA is now by far the clandestine choice. See *Practical LSD Manufacture* or *Secrets of Methamphetamine Manufacture* for details on doing the pipe bomb method. See also the chapter in this book for a convenient method of making ammonia saturated alcohol solution. The best alcohol to use in the pipe bomb method is isopropyl alcohol, of at least 91% strength, obtained at the drug store. Even better is ISO-HEET gas line de-icer, which is like 99% isopropyl alcohol. To get good results in the pipe bomb method, water should be kept out of the solution to the extent possible.

Now to details of the so-called DMSO method of making bromosafrole which Pugsly has posted. The scale given is just for example, it can be scaled up to any size desired. To 100 ml of ice cold DMSO (dimethylsulfoxide) add 7.8 ml of concentrated sulfuric acid. Mix this in well, then add 30 grams of sodium bromide, or 35 grams of potassium bromide. Both work, so use whichever is most easily obtained. Mix this solution up thoroughly. The solution will gradually turn orange colored, and crystals of sodium sulfate will form. Don't bother to filter them out, just let the process go. If the DMSO, sulfuric acid and NaBr were free of water, then the HBr formed will also be anhydrous. This is important to getting good results.

Next add 5 ml of sassafras oil, and stir it in well. Then allow the solution to warm up to room temperature. Over the course of a day or two the solution will progress through green color to purple and finally ending in a burgundy color, just as in the case of dehydrating 48% HBr with HCl gas. After a couple of days, dilute the solution with about 1000 ml of water. The bromosafrole product settles to the bottom. If you cool down the mixture, the bromosafrole becomes

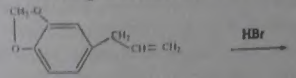
pretty sticky, so that the water and DMSO solution can just be poured off from the settled bromosafrole.

Now that's an easy recipe! Nothing stinky to give one's self away. The DMSO one could even find at a health food store or other places catering to alternative medicine. In any case it is a quite common industrial solvent. The sulfuric acid one can get from mail order chemical suppliers, as can the NaBr or KBr. Hobby shops and the local chemical "experimenters" shop can also be trusted to get these innocuous chemicals. This method is unstoppable! As to the ammonia needed to make the MDA from the bromosafrole, this too is safely ordered from the same outlets. If one doesn't want to do that, I'm told that Ace Hardware stocks a "Janitors Industrial Strength Ammonia" which is 28% NH_3 or 56% NH_4OH. This can be used to make ammonia in alcohol solution by the simple method given in this book in the chapter of that title. If MDMA or "X" is desired, just use methylamine instead. See the procedures for making methylamine in this book, and in *Secrets of Methamphetamine Manufacture*. The free base of methylamine must be used, not methylamine hydrochloride. Methylamine can also be boiled out of solution and piped into alcohol to make the methylamine/alcohol solution by the procedures given in the *Secrets of Methamphetamine Manufacture*, and in this book.

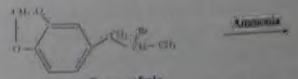
For the best results, the bromosafrole made by this method should then be dissolved in some toluene, washed with bicarb solution, then distilled under a vacuum in the usual manner for bromosafrole. See *Secrets of Methamphetamine Manufacture* or *Practical LSD Manufacture*.

Chapter Seventeen
A Convenient Method For Making Ammonia Saturated Alcohol

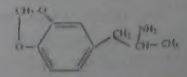
One of the better methods of making MDA, MDMA, or other psychedelic amphetamines from commonly available essential oils is to react the allylbenzene found in the essential oil with hydrobromic acid to give the bromo derivative, and then heat the bromo derivative inside a steel pipe with an alcohol solution saturated with ammonia or methylamine to produce the amphetamine. To illustrate for MDA:



Safrole
(80-90% of sassafras oil)



Bromosafrole



MDA

See the Fourth Edition of *Secrets of Methamphetamine Manufacture* for a complete discussion of the ins and outs of doing this reaction sequence. Of particular value in that book is my own procedure for using the more commonly available 48% strength HBr to produce bromosafrole. Previously published procedures used exotic reagents like 70% HBr or HBr gas.

One of the more bothersome aspects of using this reaction sequence to make MDA is the downwind smell and labor involved in making alcohol saturated with ammonia. One can't simply take the 28% strength ammonium hydroxide so easily available and pour it into alcohol to get ammonia in alcohol. The 72% of water that this ammonium hydroxide contains interferes with the reaction, producing unwanted byproducts. To get good results, the amount of water in the mixture must be kept down.

In my previous books, I suggested two methods for getting reasonably dry ammonia solutions in alcohol. Method number one, which is still the best, is to get a cylinder of ammonia at the welding supply shop (for refrigeration uses). The cylinder of anhydrous ammonia can then be held upside down, and liquid ammonia run into the alcohol by cracking open the valve.

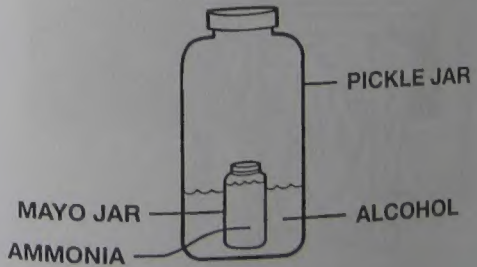
Suggested method number two, which I personally know to work well, is to pour some of the 28% NH_3 or 56% NH_4OH into a round bottom flask, attach a reflux condenser to the flask and run cold water through it.

Then the ammonium hydroxide is heated to boil out the ammonia, and the vapors of ammonia which pour out the top of the condenser are piped into some alcohol. The alcohol must be cooled and rapidly stirred to catch most of the ammonia piped into it. Still, a lot escapes to create a stench

which may cause unwelcome attention. Also a couple hours of labor are required to make a few hundred mls of ammonia saturated alcohol.

A correspondent has written with his improved method for making alcohol saturated with ammonia. Virtually no labor is involved, and grocery store equipment is used. I haven't personally tried it, but it looks really good, and should produce an alcohol product that has very little added water.

My correspondent starts with a one gallon pickle jar or other similar glass container with a wide top. After dumping out the pickles and cleaning everything up, the jar is placed on a flat surface, and a one pint mayonnaise jar or similar container is placed inside the jar. It is filled with strong ammonia solution (28% NH_3, 56% NH_4OH). Then in the space around the smaller jar inside the jug, put about a pint of alcohol. Then close the lid on the pickle jar.



In a day or two, the ammonia vapors work their way into the alcohol to the point where the two solutions reach equal concentrations, about 15%, which is about the saturation

point for alcohol. Then the mayo jar and its ammonia is removed, and the saturated alcohol is poured into a champagne bottle with a plastic stopper. It can be kept there until it is used.